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INTRODUCTION TO CHEMICAL PREPARATIONS.

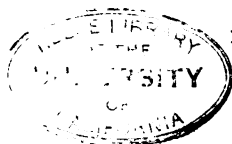
*A GUIDE IN THE PRACTICAL TEACHING
OF INORGANIC CHEMISTRY.*

BY
PROFESSOR DR. ERDMANN,
OF HALLE.

AUTHORIZED TRANSLATION FROM THE SECOND GERMAN EDITION

BY
FREDERICK L. DUNLAP, S.D.,
Instructor in Industrial Chemistry at the Worcester Polytechnic Institute.

FIRST EDITION.



FIRST THOUSAND.

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TRANSLATOR'S PREFACE.

IN presenting to the public this translation of Professor Erdmann's book, it is hoped that in this form, which is more serviceable to the English-speaking student, it may aid in advancing the recognition due so important an adjunct in laboratory instruction in chemistry as that of the subject of inorganic chemical preparations. It is gratifying to see that recognition is being given to inorganic chemical preparations, by the introduction of work of this nature among the courses of some of our educational institutions. It is particularly desirable that this subject be given a broader scope than has hitherto been afforded it; for it gives to the student a specially broad field in which to acquire chemical manipulation of a quite varied nature. Then, too, with such a wisely chosen and diversified list of experiments as Professor Erdmann has made use of, the opportunity for the observation of phenomena of different kinds lends itself admirably to the development of chemical thought in a perfectly natural way. The introduction of the subjects of qualitative and quantitative analysis

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into this work, is accomplished in such a manner that it may, with the judicious guidance of the instructor, help in preventing these subjects from degenerating into a "blind following of directions," as they not infrequently do.

The translator has left Professor Erdmann's book practically unchanged; a few foot-notes, serviceable in a German edition but less so in an English one, have been omitted. A more detailed cross-section of a Rössler gas-furnace has been substituted for the one used in the German edition.

FREDERICK L. DUNLAP.

WORCESTER, MASS., February, 1900.

PREFACE.

WHEN this introduction first appeared, it was but little customary to give the preparation of inorganic substances a significant place in laboratory instruction. It is true there never was a doubt that the chemical ideas of the student are built up only by his own observation; but only in the special domain of the carbon compounds has the logical conclusion of this been drawn. It was thought that in inorganic chemistry one was able, after a few test-tube reactions, to go directly into systematic analysis, which, in consequence, frequently degenerated into a blind following of directions. In opposition to this, I took the ground that the systematic observing and executing of various kinds of reactions on not too small a scale, was the most valuable means for the development and exercise of chemical thought, and, consequently, must constitute the basis for practical instruction. From this results the demand for selecting, from a didactic standpoint, preparations from the whole field of chemistry. To the very carefully pre-

pared, reliable, and thorough directions, analytical hints could then be added in a natural way.

My first attempt in this direction found everywhere such friendly acceptance and criticism that I gladly comply with the wish of my publishers for a new edition. The text is much improved by kind communications of my colleagues and by my own practical experience. The following preparations are added: ammonium perborate; anhydrous aluminium chloride; thorium dioxide; arsenious oxide; violet chromium sulphate; potassium iodate. The index, tables of atomic weights, etc., as also some new illustrations, will contribute to the usefulness of the guide.

In the choice of the preparations, care was taken to present to the student many fundamentally different reactions. The author trusts to have done justice to the economy of the laboratory by starting, as far as possible, with cheap or valueless materials; for example, the residues of all sorts accumulated in every laboratory, or the by-products from other preparations. The finished preparations are, on the other hand, for the most part substances which are continually used in the laboratory in analytical and synthetic processes, but which are either not generally found in the market or else not obtainable in a sufficiently pure state.

The arrangement of the guide is based on the natural system of the elements (page 114). Each of

the more important elements is represented by some characteristic compounds, so that the student who makes a preparation of each of the thirty-five elements considered here, obtains an idea of the whole field of chemistry.

The appendix treats briefly of a few of the most important helps in the laboratory, among other things of the technique of gas currents, recently so much improved.

H. ERDMANN.

HALLE a. S., April, 1899.

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INORGANIC CHEMICAL PREPARATIONS.

SODIUM AMALGAM, 10%.

Heat 3 kilograms of mercury strongly with a Berlin burner (see appendix), in a covered iron pot under a hood, and add, in rapid succession, 300 grams of sodium * cut into pieces of about 5 grams each. At each addition of sodium, the reaction must take place immediately, accompanied by a flash. If towards the end of the operation this ceases to be the case, increase the heat and stir with an iron rod. After all the sodium has been added, pour the amalgam upon an iron plate, break it into pieces, and put it into a thick-walled, well-stoppered flask while it is still warm.

REACTION: The union of the metals takes place very easily, so long as the mass is completely liquid;

* Sodium is best protected under low boiling petroleum ether (ligroïn) and not under petroleum. Sodium waste can be used quite well at the beginning of the experiment if it is washed with ligroïn and superficially dried with coarse blotting-paper. Upon pouring out the amalgam, the impurities in the sodium remain as a carbonaceous residue in the iron pot.

but when crusts of unmelted amalgam form, in consequence of too little heat, the unchanged pieces of sodium may collect upon it. This must be avoided by stirring.

TEST: Place pieces of the amalgam broken up to the size of a pea in a small weighing-bottle, and weigh 0.3–0.5 gram from it into a dry nitrometer tube. Place dilute sulphuric acid in the cup of the nitrometer and measure the hydrogen evolved upon shaking.

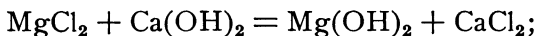
PURE SODIUM CHLORIDE, NaCl.

Triturate $\frac{1}{2}$ kilogram of common salt with $1\frac{1}{2}$ liters of cold water in a mortar of 3 liters content, filter through a creased filter into a 3-liter porcelain dish, and heat on the gas-stove. Add 5 grams of lime in the form of milk of lime, and then treat with barium chloride until there is a slight excess.* After the precipitate settles, filter into a beaker and add sodium carbonate solution (made from 15 grams of pure an-

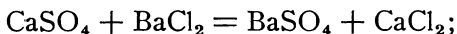
* On an average, 12 grams of crystallized barium chloride are used. As it is very difficult to recognize the end of the reaction in the turbid liquid, a small test is withdrawn, as is done in many other cases, by dipping a fine capillary tube in at the edge of the liquid where it rapidly settles. By blowing, the contents of the capillary tube are emptied upon a watch-glass. In the case in question, the watch-glass is placed on a black background and tested with a drop of barium chloride solution and with dilute sulphuric acid.

hydrous sodium carbonate). Bring this filtrate to the boiling point, neutralize with dilute hydrochloric acid (about 25 c.c.), and evaporate as rapidly as possible, by means of a large flame, to $\frac{1}{2}$ liter. Collect, in a funnel containing a platinum cone, the sodium chloride which separates; drain it by means of a filter-pump; and dry the salt by heating in a platinum or porcelain dish, stirring with a platinum spatula or glass rod.

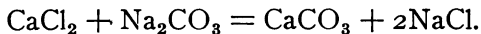
REACTION: The magnesium, as well as any iron present in the common salt, is precipitated by the milk of lime as a hydroxide:



the sulphuric acid is precipitated by means of the barium chloride:



the alkaline earths are finally separated as carbonates:



TEST: Sodium chloride consists of a snow-white crystalline powder made up of fine cubes. The dried salt should not decrepitate or give off water upon being heated in a small dry tube. At a higher temperature it should melt to a mobile fluid. Test the aqueous solution with ammonium oxalate, sodium phosphate, potassium ferrocyanide, and barium chloride, for the absence of calcium, magnesium, iron, and

sulphuric acid. One drop of the solution and ten drops of platinic chloride, evaporated on a watch-glass to a small volume, should give, upon cooling, pretty triclinic prisms of sodium chlorplatinate, $\text{Na}_2\text{PtCl}_6 + 6\text{H}_2\text{O}$, which upon dissolving in alcohol should leave no residue of potassium chlorplatinate.

SODIUM HYDROXIDE SOLUTION FREE FROM CARBONATE, $\text{NaOH} + x\text{H}_2\text{O}$.

Place 250 grams of lime (best made from marble) in a small iron pot, pour a little hot water upon the lime, and cover the vessel. Then dissolve, by heating, 1 kilogram of commercial sodium hydroxide in 3 liters of water, in an iron pot of 5-6 liters content. Make milk of lime from the lime, which has meanwhile fallen to a powder, by stirring 1 liter of hot water with it; then add this milk of lime to the boiling sodium hydroxide solution. Pour this solution into a tall flask or cylinder, which has been previously warmed by rinsing out with hot water, and close the receptacle tightly with either a stopper or a ground-glass plate. The next day, siphon the liquid, which is completely clear, into a 3-liter flask, the siphon being at first filled with water.

REACTION: Carbonic acid and silicic acid are precipitated as insoluble salts by the lime.

TEST: The sodium hydroxide solution should re-

main clear upon the addition of a drop of barium chloride solution (carbonic acid, sulphuric acid). Concerning the remaining impurities in sodium hydroxide (ammonia, lime, iron, copper, aluminium hydroxide, silica), compare with potassium hydroxide, page 9. The strength of the sodium hydroxide solution may be ascertained approximately, by means of the specific gravity, from the following table.*

Per Cent NaOH.	Specific Gravity.	Per Cent NaOH.	Specific Gravity.	Per Cent NaOH.	Specific Gravity.
10	1.115	17	1.192	24	1.296
11	1.126	18	1.202	25	1.279
12	1.137	19	1.213	26	1.290
13	1.148	20	1.225	27	1.300
14	1.159	21	1.236	28	1.310
15	1.170	22	1.247	29	1.321
16	1.181	23	1.258	30	1.332

To determine the amount of sodium hydroxide present, fill a burette with this solution and another with dilute hydrochloric acid, and determine the equivalent of both liquids (using phenolphthaleïn as an indicator) by titrating 20 c.c. of the sodium hydroxide solution with the acid, until it is colorless. Weigh 10.585 grams ($\frac{1}{10}$ mol.) of ignited pure sodium carbonate, add an excess of the dilute hydrochloric acid from the burette, heat, and then titrate back with the sodium hydroxide solution. A simple calculation

* For the specific gravity of weaker and stronger sodium hydroxide solutions, see Erdmann. *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 528.

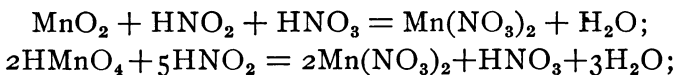
gives the strength of the acid and the sodium hydroxide. If the strength of a sodium hydroxide solution containing some carbonate is to be determined, add 5 c.c. of barium chloride solution to 20 c.c. of the sodium hydroxide solution before titrating with phenolphthaleïn as an indicator.

SODIUM NITRITE, NaNO_2 .

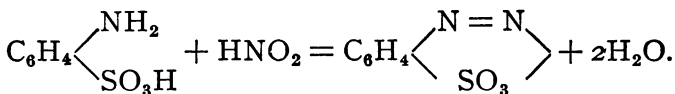
After removing the small dome from the inner fire-clay mantle of a Rössler gas-furnace, place upon the mantle a strong iron-wire triangle, and set upon the triangle a shallow iron dish ($2\frac{1}{2}$ cm. high, 12 cm. upper diameter) having a smooth bottom. Place 85 grams of Chili saltpetre in the dish and close the furnace. As soon as the dish has become faintly incandescent and the molten nitrate just begins to give off bubbles of oxygen, gradually add 206 grams of lead in the form of old pieces of sheet lead or lead tubing. The lead is at once vigorously oxidized, and, if stirred continually with an iron spatula, becomes almost completely converted into oxide of lead in half an hour. Empty the contents of the small iron dish into a large deep iron one, and repeat the operation several times, using the same amounts of Chili saltpetre and lead. Place the various products in the large iron dish, extract once with boiling water, and decant upon a creased filter. Dry the residue of lead oxide and set

aside for the experiment on page 52. Pass a strong current of carbon dioxide into the still boiling-hot filtrate, for a few minutes only; filter off the lead carbonate which separates, and neutralize the solution while stirring it, by carefully adding nitric acid from a pipette or burette. Evaporate the solution to crystallization. The crystals which separate first, consist partly of nitrate and may be used again for remelting with lead; the mother-liquor gives pure nitrite. A normal solution of the nitrite is prepared by dissolving 69 grams of it in water and diluting to one liter.

REACTION : $\text{NaNO}_3 + \text{Pb} = \text{NaNO}_2 + \text{PbO}$. A small part of the nitrite is converted into sodium plumbite, which must be decomposed by carbon dioxide.—Nitrous acid reacts with peroxides and with permanganate according to the following equations:



with sulphanilic acid it gives diazobenzenesulphonic acid:



TEST: Sodium nitrite crystals, upon treatment with dilute mineral acids, go very easily into solution, with considerable effervescence and evolution of nitrous anhydride. The acid solution turns potassium iodide

and starch paper blue and dissolves the peroxides of manganese and lead with great rapidity. To test the strength of the sodium nitrite, fill a burette with the normal solution and run it into a solution of 11.55 grams of the sodium salt of sulphanilic acid, $\text{C}_6\text{H}_4\text{NH}_2.\text{SO}_3\text{Na} + 2\text{H}_2\text{O}$, which is cooled with ice and made strongly acid with hydrochloric acid; stir constantly, and continue the addition of the nitrite solution until a drop of the liquid gives a strong blue color with potassium iodide and starch paper. If the nitrite is pure, 50 c.c. are necessary. Or, 50 c.c. of the nitrite solution may be measured into a graduated flask by means of a pipette, diluted to one liter, and this dilute solution run from a burette into tenth normal permanganate which is made acid with sulphuric acid and warmed to $40-50^\circ$, stirring during the addition. Just 50 c.c. of the twentieth normal nitrite solution should be used for 50 c.c. of the permanganate.

CHEMICALLY PURE POTASSIUM HYDROXIDE, KOH.

Literature: Schubert, *Jour. f. pract. Chem.* [2] **26**, 117.

Dissolve 300 grams of crystallized barium hydroxide in 1 liter of water in an iron dish, and add a hot concentrated solution of 120 grams of potassium sul-

phate until a test taken out by means of a capillary tube (see note on page 2) reacts with neither a potassium sulphate nor a barium hydroxide solution. Filter through a creased filter into a glass flask; and evaporate the filtrate in separate portions in a silver crucible as rapidly as possible, by means of a large flame. When the potassium hydroxide which remains is in a state of quiet fusion,* pour it into a large dish of silver, iron, or nickel, rotate it, and put the thin plates, while still hot, into a preparation bottle which is to be well-stoppered and paraffined.

REACTION: $\text{Ba}(\text{OH})_2 + \text{K}_2\text{SO}_4 = 2\text{KOH} + \text{BaSO}_4$.

TEST: The potassium hydroxide should dissolve in water and alcohol without turbidity; a solution in recently boiled distilled water should produce no precipitate upon adding a drop of barium chloride, sulphuric acid, ammonium sulphide, or Nessler's reagent. Upon heating with an excess of ammonium chloride solution in a platinum or porcelain dish until almost all the ammonia has been driven off, no precipitate should be formed (aluminium hydroxide, silica); and one should not be produced upon addition of ammonium oxalate (lime).

* In all work with molten alkaline hydroxides, it is necessary to protect the eyes by means of goggles.

PURE POTASSIUM DISULPHATE, KHSO_4 .

Literature: Krüss, *Annalen*, 238, 50.

Commercial potassium chloride is purified according to the method given for sodium chloride (page 2). Upon cooling the evaporated solution, most of the salt crystallizes out. Determine by the flame test whether or not this salt is completely free from sodium chloride. Add 20 grams of the pure potassium chloride thus obtained, by means of a platinum spoon, to a platinum dish containing 28 grams of pure, warm, concentrated sulphuric acid, and heat the dish with a small Bunsen flame until the gas evolution has completely ceased. The molten mass solidifies to a mass of large crystals.

REACTION: $\text{KCl} + \text{H}_2\text{SO}_4 = \text{HCl} + \text{KHSO}_4$.

TEST: The snow-white crystalline mass is to be tested for the absence of sodium and chlorine. Determine its melting-point, which lies somewhere above 100° .*

POTASSIUM CYANATE, KCNO .

Literature: Erdmann, *Berichte d. d. chem. Ges.* **26**, 2438, and *Chemische Präparatenkunde* (Stuttgart 1894), Bd. II, 537.

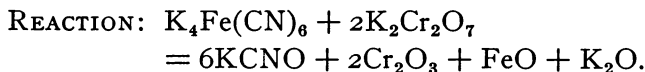
Break commercial potassium ferrocyanide into rather coarse pieces and spread it in a thin layer in

* This varies greatly, even if the salt contains only a small amount of water.

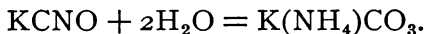
a large iron or nickel dish or pan; heat on the gas-stove with a small flame until the crystals have completely effloresced and, upon breaking open the crystals, no core of the unchanged yellow salt is present. Grind the salt while still warm to a fine powder; again spread it on the hot metal surface, and dry it at a uniform temperature for a few hours. Thoroughly mix 200 grams of the absolutely anhydrous potassium ferrocyanide thus obtained,* with 150 grams of potassium dichromate which has previously been melted and which is still warm. Add the mixture, in small spoonfuls, to a large iron dish, heated by means of a Berlin burner or gas-stove (see appendix) to such an extent that each portion of the powder, as it is added, changes to a black mass. During the addition, stir with an iron spatula. The temperature should not rise high enough to melt the mass, nor should ammonia be evolved during the operation (shows presence of water in the substances). Grind the black product from the reaction while it is still warm, and then heat on a water-bath for 10 minutes with a mixture of 900 c.c. of 80% alcohol and 100 c.c. of methyl alcohol, shaking thoroughly during the operation. Decant the clear solution through a creased filter into a large beaker which is surrounded by ice, also im-

* It is necessary that the preparation contain no trace of water, as may be determined by heating some in a test-tube, for the colorless salt which is completely effloresced retains tenaciously a small amount of water.

mediately cooling the black residue from the extraction by placing the flask in ice-water. Decant the mother-liquor from the heavy crystalline cyanate back upon the black residue, and exhaust this residue of cyanate by four or five similar extractions. Drain the various portions of cyanate on the same filter by means of a pump, wash with ether, and dry in vacuo over sulphuric acid. In this way are obtained about 85 grams of pure, dry potassium cyanate which reacts only slightly alkaline. The mother-liquor still contains potassium cyanate, which can only be obtained by evaporation in vacuo. It is better immediately to work the cyanate up into urea (page 63), as it decomposes rapidly upon standing and especially upon heating, a decomposition which makes itself evident by the odor of ammonia.



Owing to its insolubility, the potassium ferrocyanide remains behind in the process of crystallizing from alcohol. The hot solution must be rapidly cooled; for, by long heating, the water in the dilute alcohol decomposes the cyanate:



Besides the complete dryness of the raw material and the choice of a proper solvent, great rapidity in the operation of extraction is the most essential condition for obtaining a good yield of potassium cya-

nate. It is not to be recommended, therefore, to undertake this operation on a larger scale than is here specified.

TEST: The salt is analyzed by evaporating a sample with concentrated sulphuric acid and igniting the residual potassium sulphate, with the addition of a small piece of ammonium carbonate.

Behavior of Potassium Cyanate towards Solvents.	At the boiling-point there dissolves	Upon cooling there crystallizes
80% alcohol, pure	62 grams.	32 grams.
" " with 10% methyl alcohol..	76 "	37 "
" " " " acetone.....	82 "	13 "

Dissolve a few small crystals of potassium cyanate in alcohol and treat with a drop of cobalt acetate solution: the formation of the double salt, $\text{CoK}_2(\text{CNO})_4$, is recognized by the intense blue color.—Concerning the action of ammonium sulphate, see page 63.

POTASSIUM CHLORATE, KClO_3 .

Dissolve 100 grams of potassium carbonate in the smallest possible quantity of hot water in a small flask, and pass chlorine into the boiling liquid until the alkaline reaction of the solution disappears. Dilute with hot water to a volume of 350 c.c., filter, and allow it to crystallize.

REACTION:



The potassium chloride remains in the mother-liquor. Upon heating, potassium chlorate goes over into potassium chloride with the intermediate formation of potassium perchlorate, the potassium chloride volatilizing at a strong red heat.

TEST: Heat a weighed sample of the dried salt in a covered platinum crucible, first on the gas-stove and then with the small flame of a Bunsen burner, until it just reaches red heat. Avoid taking off the cover of the hot crucible.

CUPROUS CHLORIDE, CuCl .

Treat 42 grams of cupric chloride (crystallized) and 32 grams of metallic copper (granulated or turnings) with 100 c.c. of hot water, add 200 c.c. of commercial hydrochloric acid (Sp. Gr. 1.175), and gently boil on the gas-stove until colorless, the flask being nearly closed with a small funnel, through which, toward the close of the operation, add fuming hydrochloric acid. This operation lasts from one to two hours, but is substantially hastened if, for the completion of the reaction, there is added, in the form of a paste, the copper powder which is obtained as a by-product in the preparation of copper sulphate (page 16). Pour the colorless liquid from the excess

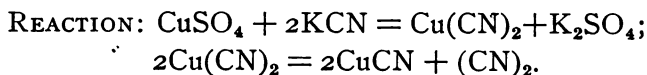
of copper into a tall cylinder filled with cold distilled water; immediately decant the curdy cuprous chloride which precipitates after it settles, drain rapidly by means of a pump, wash with alcohol and ether, and dry in vacuo over sulphuric acid.

REACTION: $\text{CuCl}_2 + \text{Cu} = 2\text{CuCl}$.

TEST: Cuprous chloride is a white, heavy substance, insoluble in water, easily oxidizing in the air, and readily going into solution by use of chlorine water or aqua regia, forming cupric chloride. Upon heating in a small tube, cuprous chloride melts without decomposition.

CUPROUS CYANIDE, CuCN .

Dissolve 50 grams of crystallized cupric sulphate in 300 c.c. of hot water; drop into the hot solution in a flask, by means of a drop-funnel, a solution of 26 grams of potassium cyanide in 50 c.c. of water. In order not to be troubled by the poisonous gas which is evolved with considerable foaming, conduct the cyanogen through an ascending tube which is drawn out to a fine point; and as soon as the apparatus is filled with the gas, burn it at the small opening. Separate the white curdy precipitate by decanting off the solution, which is still blue in color, wash it with water, and dry on a porous plate.



TEST: Cuprous cyanide is quite stable in the air; otherwise the white powder resembles cuprous chloride. It decomposes upon heating, giving off the odor of cyanogen.

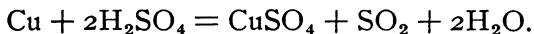
CUPRIC SULPHATE, $\text{CuSO}_4 + 5\text{H}_2\text{O}$.

Heat 200 grams of copper (granulated) in a flask on the gas-stove with 900 grams of concentrated sulphuric acid.* Wash the sulphur dioxide which is evolved with water, and dissolve in 4 liters of cold water (aqueous sulphurous acid). After cooling, pour the cupric sulphate, which remains behind as a crystalline white powder, and the sulphuric acid into a porcelain dish; rinse the flask with 1 liter of hot water, whereupon a small residue of copper which has not been acted upon is left in the flask. Treat the hot liquid with commercial nitric acid (about 25 grams) until a clear blue solution is formed; then filter through a creased filter into a dish. Dissolve the large crystals which settle out upon standing and which are somewhat white, in 300 c.c. of hot water;

* In case it is not a question of the yield of sulphurous acid, take, in place of the 200 grams of copper, 300 grams of copper scale, by means of which the yield of cupric sulphate is increased.

and by shaking in a flask and cooling with water, a fine crystalline precipitate is obtained. Filter this on a platinum cone and dry on a porous plate without warming (105 grams). If it is desired to obtain the pure product in a prettier form, crystallize again from water. Unite the mother-liquors in a porcelain dish, and treat in the cold, stirring well, with zinc dust, until the liquid is only a faint blue color. Purify the copper powder thus obtained by decantation, and preserve in the form of a paste. (See cuprous chloride, page 14.)

REACTION:



A portion of the sulphur dioxide is further reduced, forming cupric sulphide, which is again decomposed by the nitric acid that is added. From a concentrated sulphuric acid solution, cupric sulphate separates anhydrous and completely white; from dilute acid, it separates in light blue crystals which contain only a small amount of water.

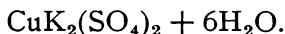
TEST: Cupric sulphate forms azure, oblique rhombohedral crystals, which slowly effloresce in the air. By heating the powdered vitriol upon the gas-stove, the completely anhydrous sulphate is obtained, which upon ignition goes over into the black oxide. Iron is tested for by means of an excess of ammonia, and zinc by precipitation with an excess of sodium hydroxide and treatment of the filtrate with ammonium sulphide.



CUPRAMMONIUM SULPHATE,



CUPRIC POTASSIUM SULPHATE,



Ammonium Double Salt.—Dissolve 30 grams of cupric sulphate in 100 c.c. of ammonium hydroxide of 0.962 specific gravity.—Place 150 c.c. of alcohol in a tall narrow cylinder; then carefully run in 20 c.c. of water underneath the alcohol, by means of a drop-funnel having a long stem; and finally add the cupric sulphate solution in a small stream, so that it collects at the bottom of the cylinder, being separated from the alcohol by the layer of water. While standing quietly from one to four weeks, large deep blue crystals form.

Potassium Double Salt.—Mix a saturated solution of 100 grams of cupric sulphate, made at a temperature of 70° , with a similar solution of 69.8 grams of potassium sulphate, the latter solution being prepared with the addition of 7 c.c. of concentrated sulphuric acid. Upon cooling, the light blue crystals of the double salt separate.

TEST: For testing, determine the amount of copper in the salts: in the case of the ammonium double salt, by simple ignition and weighing the resulting copper oxide; with the potassium double salt, by precipitation from a dilute solution, made slightly

acid with hydrochloric acid, in a small weighed platinum dish, by means of a little piece of cadmium or zinc. When a small test of the decolorized liquid no longer gives a precipitate of cupric sulphide with hydrogen sulphide, wash with hydrochloric acid, then with hot water, and finally with alcohol; dry at 100° and weigh.

PURE SILVER, Ag.

(a) *Crystalline Silver according to Stas.*—Dissolve in nitric acid any desired but known weight of silver which contains copper (coin or scrap), such as is used in the arts; filter, and evaporate to dryness. Heat the residue until it melts and begins to turn black; take up in water, filter, and dilute until the liquid contains 2% of silver. Make the cold solution strongly ammoniacal and add sufficient 40% sodium disulphite, so that a test of the blue liquid decolorizes upon heating.

Upon letting the solution stand in the cold, the greater part of the silver precipitates in beautiful crystals, the rest of it precipitating upon warming from 60° to 70° . Wash the silver with cold water, digest once with concentrated ammonia, wash again with water, dry on the water-bath, and weigh.

(b) *Molecular Silver from Residues.*—Heat silver residues with zinc bars and hydrochloric acid: sepa-

rate mechanically the reduced silver from the undissolved zinc, wash well by decantation, dissolve in nitric acid, and precipitate with hydrochloric acid. Wash the silver chloride which separates by decantation and add, while still moist, to a sodium hydrate solution which is kept boiling in a porcelain dish, adding to this hot solution from time to time a few cubic centimeters of a concentrated grape-sugar solution, until a sample of the gray silver powder, which has been filtered and washed, dissolves clear in nitric acid. Wash, dry, and weigh as under (a).

REACTION: The black color in the melting of the impure nitrate comes from the beginning of the decomposition of the cupric nitrate. In order to avoid loss of silver, do not heat so long that all the copper salt is converted into oxide; but base the separation of the silver from the copper on the fact that silver is precipitated in a metallic form from an ammoniacal solution by means of sulphurous acid, while the copper is, under these conditions, reduced to cuprous oxide which remains in solution in the ammoniacal liquid, and the slow precipitation of the silver in a crystalline form is brought about.

TEST: Dissolve a sample of the silver in nitric acid which is free from chlorine, and examine any residue there may happen to be (gold, silver chloride, stannic oxide): precipitate a solution with hydrochloric acid, and test the filtrate for heavy metals.

PURE GOLD, Au.

Gold, such as is used in the arts (broken pieces of jewelry, a coin, or crude gold dust), is cut up into as small pieces as possible, weighed, and treated in a small flask with concentrated hydrochloric acid. Heat the small flask on a sand-bath, and add concentrated nitric acid from time to time. As soon as all has gone into solution, evaporate on a water-bath, with the careful exclusion of dust, to such an extent that the remaining dark-red liquid solidifies upon cooling. Then take up in much water, filter from the silver chloride which has separated, and treat the filtrate hot with a great excess of ferrous chloride. Decant the pulverulent gold which separates, heat up once with dilute hydrochloric acid, collect on an ashless filter, heat in a porcelain crucible, and weigh.

In case larger amounts of gold are separated, a little platinum and palladium (also thallium) may be obtained from the filtrate by means of iron or zinc.

TEST: The gold, which is obtained in the form of a reddish powder, should impart no trace of a yellow color (iron) to boiling concentrated hydrochloric acid. If a weighed amount is again subjected to the procedure for separation described above, the weight must remain absolutely constant.

ANHYDROUS MAGNESIUM CHLORIDE, MgCl_2 .

Dissolve 500 grams of crystallized magnesium chloride with 500 grams of ammonium chloride in the smallest quantity of water, filter and evaporate in a porcelain dish to dryness; the last operation being best accomplished in a silver dish. Break up the solid mass while it is still hot, and dry in small portions in a porcelain dish, or, better still, in a platinum dish (all the baser metals, including silver, are attacked by this mixture at higher temperatures). This operation lasts a long time and is to be carried out with special care; for if but very small quantities of water remain behind, the successful result of the experiment is frustrated. Utilize several gas-stoves at the same time for the drying, and regulate the flames so that ammonium chloride vapors are not evolved. Grind up the portions from time to time in a hot mortar, continue the drying until the product gives a powder which does not lump together upon heating; consider the operation ended only when a sample in a test-tube gives off no moisture upon heating, and when, after driving off the ammonium chloride, there remains a quite mobile clear liquid which, upon cooling, solidifies in radiating crystals. Add the powder *

* If the dried substance is to be preserved, this must be done

while still hot to a large platinum crucible, close it with a well-fitting cover, and place it in a Rössler furnace (see appendix) which has previously been heated. After driving off the ammonium chloride, take out the crucible,* fill it again with the powder, and continue the ignition until the whole mass has been melted. The solidified magnesium chloride, which is easily removed from the crucible, is filled while still warm into a well-stoppered bottle.

REACTION: Crystallized magnesium chloride loses much hydrochloric acid upon heating; on the other hand, the double salt $(\text{NH}_4)_2\text{MgCl}_4$ is, by careful heating, dehydrated without decomposition. Upon igniting this double salt, anhydrous magnesium chloride remains: $(\text{NH}_4)_2\text{MgCl}_4 = \text{MgCl}_2 + 2\text{NH}_4\text{Cl}$.

TEST: The anhydrous magnesium chloride should form large radiating crystals of a silky lustre, not unlike those of fused sodium acetate, which melt easily upon heating and volatilize at a bright heat. The vapors decompose aqueous vapor instantaneously, forming hydrochloric acid. The very hygroscopic salt should dissolve clear in water, with the evolution of heat.

in well-stoppered bottles. Before being used, the mass is to be dried again for a short time on the gas-stove.

* When the crucible is at a bright heat, it should not be opened, on account of the volatility of the magnesium chloride.

POROUS CALCIUM CHLORIDE, CaCl_2 .

Heat 1 liter of the mother-liquor, from the generation of carbon dioxide from marble and commercial hydrochloric acid, which no longer reacts with an excess of marble; then treat it in a 2-liter iron pot with milk of lime (made from 35 grams of lime) and, after boiling, filter the strongly alkaline liquid.* Add hydrochloric acid to the filtrate until strongly acid, and evaporate in portions in a $\frac{1}{2}$ -liter porcelain dish on the gas-stove (see appendix). As soon as a layer of salt forms on the surface, turn down the flame, add some fuming hydrochloric acid, and let the solution stand quietly without stirring, so that the mass does not become fully dry until after 4-5 hours. By the aid of a knife, carefully loosen the porous calcium chloride from the dish, break up the large pieces while still hot in a warmed mortar, sort the small pieces as rapidly as possible, according to the size of the grains, by use of a sieve having meshes from 1-5 mm. in diameter, and fill immediately into a well-stoppered preparation bottle.

REACTION: The lime precipitates the magnesium, as well as the iron and manganese, from the solution

* In place of starting with this, the residue of calcium chloride and excess of lime from the preparation of ammonia may be used. Heat this up with water until the last traces of ammonia have been driven off, filter, and continue the preparation as described above.

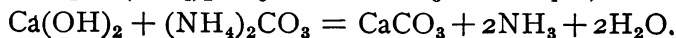
obtained from the marble. (Upon standing, basic calcium chloride, $3\text{CaO}, \text{CaCl}_2 + 15\text{H}_2\text{O}$, frequently separates from the filtrate in beautiful long prisms.) In evaporating to dryness, the solution must be acid with hydrochloric acid and the heating gradual, because otherwise the calcium chloride loses hydrochloric acid and reacts strongly alkaline.

TEST: The calcium chloride forms pure white, porous, very hygroscopic pieces. These should dissolve in water, and also in alcohol, to a clear solution; the aqueous solution should react but weakly alkaline.

CALCIUM CARBONATE, CaCO_3 .

Purify $1\frac{1}{2}$ liters of calcium chloride mother-liquors with milk of lime as described above. Pour the filtrate, while still hot, into $2\frac{1}{2}$ liters of a 10% solution of commercial ammonium carbonate containing 250 c.c. of dilute ammonia (1 part of concentrated ammonia, 2 parts of water). Decant the thoroughly mixed liquids into a tall glass cylinder, finally drain the precipitate on a filter by means of a pump, and completely dry in a nickel dish on the gas-stove.

REACTION:



TEST: The calcium carbonate is a snow-white, crystalline, tasteless powder, which dissolves clear in dilute hydrochloric acid, with strong effervescence. The hydrochloric acid solution should give no precipitate with ammonia (iron, aluminium). After the precipitation of the calcium by means of ammonium oxalate, test the clear ammoniacal solution for magnesium, with sodium phosphate. A nitric acid solution must remain clear upon the addition of silver nitrate. The preparation, when heated in a small dry tube, should give off neither water nor ammonia.

ZINC FREE FROM ARSENIC, Zn.

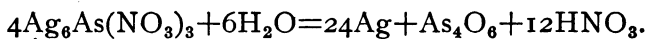
Literature: L. L'Hôte, *Ann. de chimie et de physique* [6] **3**, 141; *Compt. rend.* **98**, 1491.

Melt 1 kilogram of commercial zinc in a Hessian crucible. Introduce into the glowing metal, clear to the bottom of the crucible, pieces of anhydrous magnesium chloride (see page 22) and hold them there until they have gone into solution. In order to hold the specifically lighter chloride under the molten metal, use an iron wire of which the lower part is wound into a compact spiral. Fasten the magnesium chloride in the small bell made in this manner, by means of fine wire, and then introduce the wire into the metal. After 15 grams of chloride have been

added, let the crucible cool to such an extent that, upon uncovering the crucible, the metal no longer glows, but is still just fluid; then pour the thinnest possible stream into a pail of water. Take out the granulated zinc, drain it, and dry by warming.

REACTION: $3\text{MgCl}_2 + 2\text{As} = 3\text{Mg} + 2\text{AsCl}_3$. The arsenious chloride and the undecomposed magnesium chloride volatilize in thick clouds.

TEST: The zinc obtained in this way contains magnesium and, consequently, in contrast to chemically pure zinc, is very easily soluble in dilute acids. In order to test it for arsenic, treat in a small test-tube with dilute hydrochloric acid (1:1) and cover the tube with a small filter, the point of which is moistened with a drop of concentrated silver nitrate solution (1:1). After a greater or less length of time, yellow arsenic silver nitrate, $\text{Ag}_6\text{As}(\text{NO}_3)_3$, is formed, which decomposes with water and turns black, according to the equation:

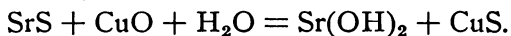
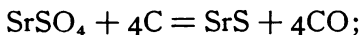


STRONTIUM HYDROXIDE, $\text{Sr}(\text{OH})_2 + 8\text{H}_2\text{O}$.

Pack an intimate mixture of 150 grams of finely pulverized celestite and 50 grams of charcoal tightly in a Hessian crucible, and cover with a layer of powdered charcoal. Close the crucible with a well-fitting

iron cover that has an attached rim, and heat at a white heat for one hour in the Rössler gas-furnace (see appendix). The cooled product must go into solution upon treatment with dilute hydrochloric acid, with the strong evolution of hydrogen sulphide, leaving but a small carbonaceous residue. Determine this point by a small test. Suspend the mass in a liter of hot water in a porcelain dish, and add well-ignited copper scale (100–150 grams) to the boiling liquid, until a filtered test no longer gives a brownish black precipitate with lead acetate. Filter hot into a beaker, heat up the residue again with 100 c.c. of water; carefully cover the combined filtrates, and let crystallize. After 24 hours, pour off the liquor from the crystals which have separated, dry these as rapidly as possible on a covered porous plate, and put them into a bottle which is to be corked and paraffined. Rapidly evaporate the mother-liquor to 300 c.c., in order to obtain a second crystallization.

REACTION: The strontium sulphate is converted into strontium sulphide by igniting with charcoal, when the air is excluded; the strontium sulphide is changed to strontium hydroxide by means of the copper oxide:



TEST: Strontium hydroxide forms transparent, efflorescent quadratic plates or needles, having a

strong alkaline taste, and is easily soluble in hot water which has previously been boiled, leaving no residue. It melts easily upon heating, and upon further heating resolidifies to a white mass, with the loss of its 8 molecules of crystal water. At a still higher temperature it again melts and is finally converted into strontium oxide. An acetic acid solution of the strontium hydroxide should not be precipitated by potassium dichromate (barium), but the strontium should be so completely removed by sulphuric acid that ammonium oxalate gives no precipitate in the filtrate (calcium).

CADMIUM CARBONATE, CdCO_3 .

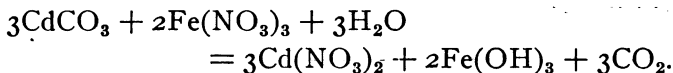
Place 100 grams of commercial cadmium, which has been cut into pieces or granulated, in a flask with 400 c.c. of cold nitric acid of a specific gravity of 1.2; leading the oxides of nitrogen, which are evolved in large amounts, into the flue of a hood by means of an ascending tube. After the action has completely ceased, pour the liquid from the undissolved portion into a porcelain dish of 6 liters content, dilute with 4 liters of hot water, and add commercial ammonium carbonate in the form of a powder, until there is just a small permanent precipitate formed. Filter this off, and, by the addition of an excess of ammonium carbonate (about 150 grams), precipitate the pure

cadmium carbonate from the filtrate. Decant several times with hot water in a tall glass cylinder, and preserve the carbonate as a paste or dry it upon the water-bath.

REACTION:



The metals present as impurities, with the exception of the zinc and iron, do not remain in solution, but are again thrown out in a metallic form by the excess of cadmium. The iron precipitates with the first portion of the cadmium carbonate:



The zinc is kept in solution by the excess of the ammonium salt.

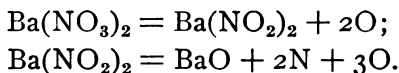
TEST: Cadmium carbonate is a pure white salt, which forms brown cadmium oxide upon ignition and dissolves readily, with effervescence, in dilute acids. It is to be especially tested for antimony, bismuth, copper, lead, iron, and zinc, according to the customary methods of analysis.

BARIUM OXIDE, BaO.

Reduce 150 grams of heavy spar with 40 grams of charcoal, according to the method described for celestite (page 27), and decompose the barium

sulphide thus obtained, in a liter flask by means of dilute nitric acid, the flask being fitted with a drop-funnel and a tube for conducting away the gases evolved. Filter the solution and evaporate to crystallization. Add $1\frac{1}{2}$ kilograms of the salt thus obtained, or commercial barium nitrate, gradually, by means of an iron spoon, to a white-hot Hessian crucible which is kept covered with a fire-clay cover that fits as well as possible. After the final addition, heat the closed crucible for an hour at the highest possible temperature. After it is partially cooled, remove the upper layer, which is colored green (principally through the presence of barium manganate), and fill the barium oxide immediately into a small flask which is closed by being sealed off.

REACTION: Upon ignition, barium nitrate is converted first of all into barium nitrite and then into barium oxide:



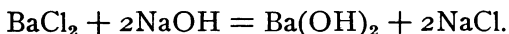
TEST: Barium oxide forms grayish-white porous pieces that heat up with water and dissolve clear in dilute acids without effervescing. This solution should not decolorize permanganate, or turn potassium iodide and starch paper blue (barium nitrite and barium peroxide).

BARIUM HYDROXIDE, $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$.

This preparation may be made from barium sulphide, according to the method given for strontium hydroxide, or in the following way:

Dissolve 1 kilogram of barium chloride in 2 liters of hot water, add 1115 grams of sodium hydroxide solution of 33° Bé. (or specific gravity 1.29),* filter, and set aside to crystallize. From hot water twice recrystallize the barium hydroxide, which is filtered off on a platinum cone by means of a water vacuum pump, and dry and preserve in the same way as the strontium hydroxide.

REACTION:



TEST: The barium is completely precipitated from an acetic acid solution by the addition of potassium dichromate. The filtrate, the color of which must be somewhat darker yellow than that of the barium chromate, should be precipitated neither by ammonium oxalate (calcium) nor by dilute sulphuric acid upon long standing (strontium).

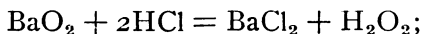
* To prepare this, dissolve 310 grams of commercial sodium hydrate in 840 grams of water.—See also pages 4 and 5.

BARIUM PEROXIDE, BaO_2 ;
HYDRATED BARIUM PEROXIDE,
 $\text{Ba}(\text{OH})_4 + 6\text{H}_2\text{O}$.

(1) Place 30 grams of barium oxide in a hard glass tube, and heat moderately in a combustion-furnace, in a strong current of oxygen which has been dried by concentrated sulphuric acid. Add the peroxide, which has been cooled and ground up with a little water, to 50 c.c. of dilute hydrochloric acid which is cooled to 0° . Filter the still weakly acid solution, and add it to 500 c.c. of saturated barium hydrate solution which is cooled to 0° . Filter the precipitate, which consists of small shining crystalline leaflets, wash with ice-water, and preserve as a paste.

(2) Cool $\frac{1}{2}$ liter of commercial hydrogen peroxide in a flask to 0° , and treat with a cold saturated barium hydrate solution until there is formed a small precipitate which does not dissolve and the solution is alkaline. Cool the filtered solution further and pour, with constant stirring, into 2 liters of ice-cold barium hydrate solution. When the shining crystalline precipitate does not increase any more in bulk, filter off the hydrated barium peroxide and wash with a little ice-water.

REACTION: At a moderate heat, barium oxide adds another atom of oxygen. Barium peroxide is decomposed by hydrochloric acid:



the hydrogen peroxide and barium hydroxide react as follows:



TEST: Upon triturating with water, barium peroxide should not heat up, showing the absence of barium oxide). The quality of the hydrated peroxide is shown if hydrogen peroxide is made from it, according to the method on page 78.

DISTILLED MERCURY, Hg.

Literature: H. N. Morse, *American Chemical Journal* **7**, 60.

In a combustion furnace, one end of which is 15 mm. higher than the other, place a hard glass tube, both ends of which are bent downwards as illustrated in Figure 1. One end of the tube, which is drawn out, is attached to a fairly thick-walled glass tube, of about 10 mm. outside diameter, by means of a rubber connection of pressure tubing, the end of the glass tube dipping into a narrow glass cylinder. The length of the glass tube is such that the total vertical height, up to the opening of the tube where it begins to slant upwards, is about 700 mm. The other end of the combustion tube is bent somewhat upward within the furnace, so that the last flame of the furnace stands under the highest portion of the tube.* From this

* Place in this part of the tube a loose plug of asbestos.

point the tube extends downwards for 20 cm. without being contracted; into this end of it is introduced a

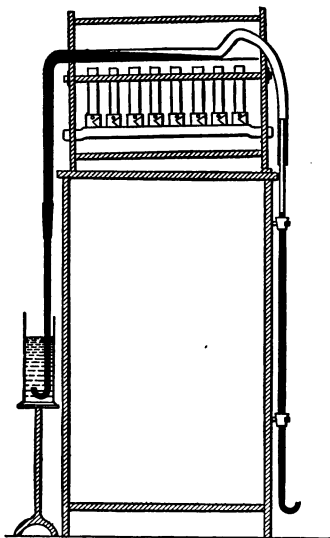


FIGURE 1.—Distillation of Mercury in Vacuo.

second glass tube, which, like the tube attached at the other end, is bent into a hook at the bottom, being held in position by means of a piece of rubber tubing.* At least 5 cm. of the tube, which is introduced into the wider one, is free, its total length being about 80 cm. In order to start the furnace, pour into the cylinder commercial mercury, or mercury which, on

* In order to be surer of a completely air-tight joint at this place, utilize the arrangement as drawn in Figure 2. This device, which surrounds the inner tube, is made by means of a rubber stopper and a small piece of large glass tubing, the cup formed being filled with mercury.

account of its use with others metals, has become impure. Exhaust the air by means of a good water vacuum pump, placing an empty Woulff bottle between the apparatus and the pump. When the mercury has risen high enough to form a layer of the liquid throughout the length of that part of the com-



FIGURE 2.—Air-tight Mercury Trap.

bustion tube lying within the furnace, the tube leading to the pump is closed with a pinch-cock. Heat the combustion tube with a small flame, so that the mercury just barely boils and distils through the asbestos plug. As soon as the descending tube is half-filled with mercury, let the air carefully into the pressure tubing by opening the pinch-cock, and remove the pressure tubing; for the mercury column prevents the entrance of air into this side of the apparatus. The apparatus now works automatically and the distilled mercury falls continuously into a receiver, if the requisite amount of mercury is placed in the cylinder from time to time.

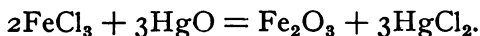
TEST: Mercury should have the specific gravity of 13.595 at 0° , and be of a pure silvery lustre; it should be mobile and form, upon being poured out, only spherical drops, leaving no oblong, thread-like residue of a more viscous character. Five grams heated in a porcelain crucible must leave no weighable residue after ignition.

MERCURIC CHLORIDE, HgCl_2 (from mercury residues).

Subject residues, which have been oxidized with commercial hydrochloric and nitric acids and evaporated to dryness on a water-bath in a porcelain dish covered with a large funnel, to a slow sublimation on a bath of sand or iron filings. According to its purity, dissolve $\frac{1}{10}$ to $\frac{1}{5}$ of this sublimate in hot water, precipitate with sodium hydrate, and, after washing and drying the precipitate, grind, with the main portion of the sublimate, the mercuric oxide thus obtained. Place in a flask the resultant powder, which is blackened by the formation of mercuric oxychloride, cover loosely with a watch-glass, and heat with a small flame on a bath of iron filings. The sublimate deposits in the upper portion of the flask in very beautiful long crystals. When the sublimation is finished, take the flask from the bath, crack the bottom of it by passing a moist sponge rapidly over it, and separate the crys-

tals from the glass, on a large sheet of paper, by means of a feather. If less well-crystallized portions are also found, these are recrystallized from 4 parts of boiling water.

REACTION: The crude sublimate is contaminated by other volatile metallic chlorides, which are all decomposed by mercuric oxide; for example:



TEST: Mercuric chloride forms snow-white, rhombic prisms, which are easily soluble in cold alcohol and ether, also in hot water, leaving no residue (calomel, ferric oxide, etc.), and which completely volatilize upon heating in a test-tube.

MERCURIC CYANIDE, $\text{Hg}(\text{CN})_2$.

Treat a dilute solution of hydrocyanic acid (see page 62) in a flask with yellow mercuric oxide, until the solution reacts strongly alkaline to litmus paper. Add sufficient hydrocyanic acid to the filtrate, so that it gives off the odor of it and reacts distinctly acid; then evaporate to crystallization. Before again evaporating the mother-liquor to crystallization, add some more hydrocyanic acid.

TEST: Mercuric cyanide is soluble in 11 parts of cold water, reacts neutral, and forms quadratic columns and pyramids which have an unpleasant metallic

taste. On heating in a small tube, the salt evolves cyanogen, forms a sublimate of small drops of mercury, and leaves a black residue of paracyanogen.

MERCURIC SULPHIDE, HgS .

Grind 60 grams of mercury and 23 grams of flowers of sulphur together in a mortar, until no more mercury is to be seen; treat with a solution of 15 grams of potassium hydrate in 80 c.c. of water, and digest at about 45° for several days, stirring frequently and replacing the water lost by evaporation. When the mass has become red, it is separated by levigation from the unchanged mercury and the greater part of the sulphur, heated with a sodium sulphite solution, filtered, and washed with hot water. About 40 grams of mercuric sulphide are obtained.

TEST: Mercuric sulphide is a scarlet-red powder which, on strongly heating in a small tube, completely volatilizes, leaving no residue.

BORON, B.

Literature: Erdmann, *Lehrbuch der anorganischen Chemie*, page 403.

Mix thoroughly, while still warm, 100 grams of borax, which has been melted and then finely powdered, with 50 grams of magnesium powder; place

the mass in a Hessian crucible, pack tightly, and cover with a layer of pure borax. All these operations must be carried out as rapidly as possible, using warm apparatus; for the dehydrated borax is very hygroscopic. Close the apparatus with a specially constructed well-fitting cover which is made of sheet iron and has a flange fitting over the outside of the crucible; heat in a Rössler furnace (see appendix) for a quarter of an hour at a red heat. After cooling, pulverize the product of the reaction, heat with water, then with hydrochloric acid, and finally again with water; and dry the resulting gray-brown powder on the water-bath.

BORIC ACID, H_3BO_3 .

Dissolve 200 grams of pulverized borax in $\frac{1}{2}$ liter of boiling water, and add 300 c.c. of dilute hydrochloric acid. After a few days, filter, on a platinum cone, the flaky crystals which separate, wash them with a very little water and recrystallize from water.

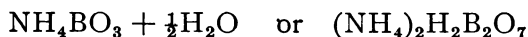
TEST: The white crystals, which have a mother-of-pearl lustre, should dissolve in 6 parts of alcohol and 25 parts of cold water. They should melt upon heating, with loss of water, to a colorless bead, which, upon cooling, forms a transparent glass-like mass. The aqueous solution reddens litmus paper; and if strips of turmeric-paper are saturated with this solu-

tion and dried in a warm place, the yellow dyestuff is changed to an orange-brown, the latter color changing to green-black with alkalis. Of impurities, hydrochloric acid and sulphuric acid are particularly to be tested for.

AMMONIUM PERBORATE, $\text{NH}_4\text{BO}_3 + \frac{1}{2}\text{H}_2\text{O}$.

Literature: Melikoff and Pissarjewsky, *Berichte d. d. chem. Ges.* **31**, 953; *Zeitschr. anorgan. Chem.* **18**, 62.

Add to 500 c.c. of a 3% hydrogen peroxide solution, which is cooled externally with ice-water, 25 grams of finely pulverized boric acid, the content of the hydrogen peroxide solution having been determined, shortly before its use, by titration with permanganate (see page 80). After the acid has gone into solution, make the solution alkaline with 30 c.c. of concentrated ammonia (specific gravity 0.89) and precipitate the ammonium perborate by the addition of $\frac{1}{2}$ liter of alcohol. Filter off the white crystals and dry them in vacuo over sulphuric acid. In a freshly precipitated condition, the salt contains 3 molecules of water of crystallization; but after standing one day over sulphuric acid, this unstable salt, which is rich in water, goes over into the more stable compound



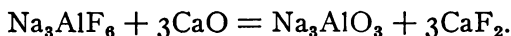
(analogous to sodium metantimonate, $\text{Na}_2\text{H}_2\text{SbO}_7$).

TEST: Treat 0.15 gram of the salt in a test-tube with 5 c.c. of lukewarm water. The salt dissolves slowly, giving off the odor of ammonia and evolving oxygen; ammonium nitrite is formed at the same time. A solution of the ammonium perborate acidified with sulphuric acid, turns potassium iodide and starch paper blue; it contains hydrogen peroxide and decomposes very rapidly. The salt evolves chlorine with hydrochloric acid, and ozone with concentrated sulphuric acid.

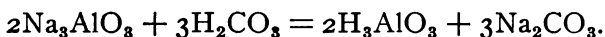
ALUMINIUM HYDROXIDE, $\text{Al}(\text{OH})_3$ (from cryolite).

Finely pulverize 50 grams of cryolite, mix intimately with 50 grams of lime made from marble, and ignite strongly in a platinum crucible for half an hour in a Rössler furnace (see appendix). Heat the pulverized frit with water; remove the traces of lime that may happen to be present in the filtrate, by means of a few drops of sodium carbonate solution; and then precipitate the solution hot, by means of carbon dioxide. Decant, with much hot water, the aluminium hydroxide which separates, drain, bring to dryness on the water-bath, and, if desirable, convert into anhydrous aluminium oxide by ignition. By evaporating the filtrate, pure sodium carbonate is obtained.

REACTION: The sodium aluminium fluoride is decomposed by the lime, forming sodium aluminate, which is soluble in water:

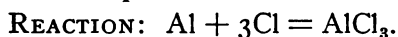


The sodium aluminate is decomposed by the carbonic acid:



ANHYDROUS ALUMINIUM CHLORIDE, AlCl_3 .

Wash 50 grams of aluminium turnings with alcohol and ether, dry them, and gently heat them in a retort in a stream of dry chlorine. The chloride condenses in the neck of the retort in colorless hexagonal plates, or as a compact laminated mass.



Anhydrous aluminium chloride is an exceptionally reactive substance, which gives compounds with the alkaline chlorides, ammonia, hydrogen phosphide, sulphur dioxide, and the chlorides and oxychlorides of phosphorus, nitrogen, and sulphur, as well as with many hydrocarbons. It serves in organic synthesis as an energetic agent for producing condensations.

TEST: Aluminium chloride fumes in the air, is decomposed immediately by the moisture of the air into aluminium hydroxide and hydrochloric acid gas, and

dissolves in water with a hissing noise and the production of much heat, the same phenomena as in the case of phosphorus pentachloride. It is not known what chemical changes take place under these circumstances. Upon cooling or evaporating in vacuo, columnar crystals of an unknown constitution are obtained, which have the empirical constitution $\text{AlCl}_3 + 6\text{H}_2\text{O}$. Upon heating, these crystals do not break down into water and aluminium chloride, but into hydrochloric acid, water, and aluminium hydroxide.

PURE OXALIC ACID, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$.

Treat 1 kilogram of commercial oxalic acid with 700 c.c. of hot water, and heat for a few minutes. Pour off from the undissolved portion, and add 300 c.c. of fuming hydrochloric acid to the liquid. After the acid, which separates upon cooling, has been washed with a very little cold water, recrystallize it from boiling water.

REACTION: The oxalates mixed with the commercial oxalic acid are less soluble than it, and, for the most part, remain undissolved upon treatment with an insufficient amount of water. The rest of the compounds are decomposed by the strong acid.

TEST: 5 grams of this preparation, volatilized in a weighed platinum dish, should leave no weighable residue.

ANHYDROUS OXALIC ACID, $\text{H}_2\text{C}_2\text{O}_4$.

Divide 500 grams of commercial oxalic acid among a large number of pasteboard boxes, spread out the acid in thin layers in them, and dry in a steam-drying closet, whereby the portion of the paper in contact with the anhydrous acid is partially carbonized. Sublime the crystals, which have completely crumbled, on a sand-bath at $150\text{--}160^\circ$. The anhydrous oxalic acid forms very long, white, completely ashless crystals, which take up moisture rapidly from the air and consequently increase considerably in weight. Upon warming, organic chlorides are energetically decomposed by anhydrous oxalic acid, with the splitting off of hydrochloric acid; benzalchloride thus produces benzaldehyde:

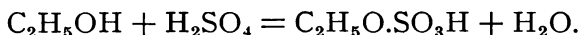
ETHYL BROMIDE, $\text{C}_2\text{H}_5\text{Br}$.

Literature: Erdmann, *Chemische Präparatenkunde* (Stuttgart 1894) Bd. II, 37.

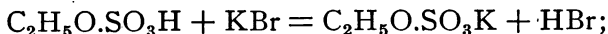
Quickly add 218 c.c. of sulphuric acid of a specific gravity of 1.835, to 218 c.c. of 96% alcohol in a dish, stirring rapidly during the addition. After cooling, add 150 c.c. of ice-water, stirring carefully; then add the mixture to 200 grams of potassium bromide in

a 2-liter flask. Fit the flask with a three-hole rubber stopper, carrying an ascending tube, thermometer, and a bent tube connected with a condenser. The condenser discharges into an adapter, which dips vertically into a receiver containing some water. The reaction is started by heating slowly on the gas-stove. It takes place mostly between 90° and 110° , but at the end the temperature must be raised to 125° . Separate carefully from the water the ethyl bromide thus obtained, cool in a cooling mixture, and add concentrated sulphuric acid, drop by drop, until the acid layer falls to the bottom as a heavy oil. After removing the sulphuric acid, which takes up the ethyl ether mixed with the ethyl bromide, wash with water, dry over calcium chloride, and rectify on the water-bath. Catch the very volatile distillate in a receiver cooled by ice. Yield, about 150 grams.

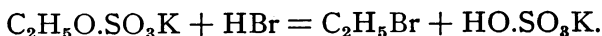
REACTION: Ethyl sulphuric acid is formed by the rapid mixing of the alcohol and sulphuric acid, much heat being evolved:



This is decomposed by the potassium bromide, forming potassium ethyl sulphate, according to the equation:



but this salt is broken up by the hydrobromic acid which is formed:



The addition of water, as described, is very important for the success of the operation; for, if omitted, the hydrobromic acid is evolved as a gas before it can enter into the reaction and much ethyl ether is formed.

TEST: The preparation should react neutral, have a boiling-point of $38-39^{\circ}$, and a specific gravity of 1.476. If a smaller specific gravity is observed, the product contains ethyl ether, which is removed by shaking out again with a half to an equal volume of concentrated sulphuric acid. The odor is quite pleasant, refreshing, and fruity; and upon rubbing the liquid between the hands, there should be left no trace of a less pleasant, or garlic-like odor. The product which is bought, is frequently made by means of commercial phosphorus, and contains, consequently, volatile arsenic compounds that may occasion poisoning. Examine the commercial ethyl bromide by burning a few cubic centimeters in a small spirit-lamp, aspirating the products of combustion through sodium hydroxide, and testing the latter for arsenic by means of stannous chloride and hydrochloric acid.

CRYSTALLINE SILICON, Si.

Literature: Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), pages 484 and 495.

Mix 50 grams of dry magnesium powder with 200 grams of very dry sea-sand (fine round-grained quartz

sand), and divide the mixture among 5 test-tubes. The tubes are held successively in an iron clamp and heated with a blast-lamp, beginning at the base and heating upward. Heat until each portion has reacted, as shown by its glowing. Pulverize the impure amorphous silicon thus obtained,* and heat with ten times its weight of zinc, in a Hessian crucible, until the zinc just begins to volatilize. Wash the zinc regulus with water and dissolve in hydrochloric acid, whereupon the silicon remains as dark, shining, regular crystals. Wash the crystals with water, warm with nitric acid, and finally heat with water again.

REACTION: $\text{SiO}_2 + 2\text{Mg} = \text{Si} + 2\text{MgO}$. The silicon is crystallized from the molten zinc, leaving the admixture of magnesia undissolved.

SILICON TETRACHLORIDE, SiCl_4 .

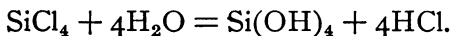
Fill a hard glass tube of 2 cm. diameter half full of the impure amorphous silicon which contains magnesia, as prepared above; place horizontally in a combustion furnace, and, while gently heated, conduct a stream of dry chlorine through it. Condense the silicon tetrachloride which distils over, in a receiver surrounded with a cooling mixture of ice and salt, and

* The fragments of the test-tubes are to be freed immediately from the magnesium silicide present (from which there is danger of fire), by treating with hydrochloric acid.

take up the excess of chlorine in sodium hydrate. Shake the greenish liquid with mercury and distil on a water-bath, using a condenser; and have the bulb of the thermometer immersed in the liquid.

REACTION: $\text{Si} + 4\text{Cl} = \text{SiCl}_4$. The free chlorine dissolved in the silicon tetrachloride is taken up by the mercury upon shaking.

TEST: Silicon tetrachloride should boil at $58-60^\circ$ and form a colorless, quite mobile liquid, which fumes strongly in the air and which decomposes immediately with water, forming orthosilicic acid:



ANHYDROUS STANNOUS CHLORIDE, SnCl_2 .

Heat commercial stannous chloride slowly on the gas-stove; the salt melts in its water of crystallization, becomes pasty, and gradually solidifies. At the end of the operation, remelt the dehydrated salt. After cooling, put it into a retort of difficultly fusible glass, the upper part of which, to avoid too much radiation, is provided with a hood of wire netting or asbestos. Distil as rapidly as possible into a porcelain dish which is kept covered with another one. As the stannous chloride begins to boil at 610° , the heating must be very strong; and as a source of heat, it is best to use a small blast-flame. Heat the neck of the

retort with a Bunsen burner, in order that the neck may not become stopped up with the solidified chloride.

TEST: Stannous chloride is a white crystalline body which melts at 250° and is easily soluble in water, alcohol, ether, and sodium hydrate.

ANHYDROUS STANNIC CHLORIDE, SnCl_4 .

Spiritus fumans Labavii.

Literature: Lorenz, Zeitschr. anorgan. Chem. **10**, 44; Erdmann, Lehrbuch der anorganischen Chemie, page 658.

For the preparation of stannic chloride, use the apparatus illustrated in Figure 3. Fill with dry granulated tin the tube *A*, which is 6 cm. wide and 100 cm. high, to *a*; add a small amount of stannic chloride (to *b*), and then pass in chlorine, which is dried by means of concentrated sulphuric acid.* The chlorine is readily absorbed by the tin tetrachloride, the liquid soon turning yellowish-green; then the action upon the metal begins with spontaneous heating, the volume of the liquid rapidly increases, while the pieces of granulated tin assume a corroded appearance and finally mat together. When all the tin changes to chloride, rectify over tin-foil in a fractioning flask, hav-

* Relative to the evolution of chlorine and the absorption of the gases which are evolved, see the appendix.

ing the bulb of the thermometer immersed in the liquid.

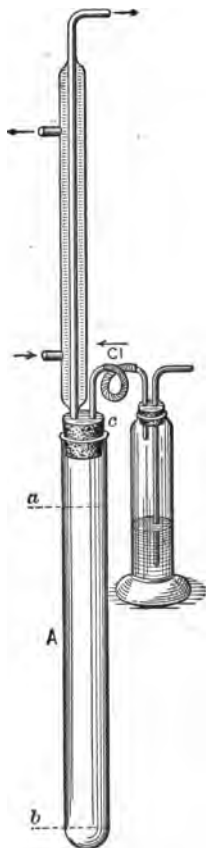


FIGURE 3.—Preparation of Anhydrous Stannic Chloride.

REACTION: $\text{Sn} + 4\text{Cl} = \text{SnCl}_4$. The free chlorine present is taken up during the fractioning by the metal which has been added.

TEST: Stannic chloride is a colorless liquid which fumes strongly in the air, boils at 114° , and has a specific gravity of 2.278 at 0° . It is easily soluble in organic liquids (oil of turpentine, carbon disulphide). Stannic chloride also dissolves clear in water, with contraction and evolution of much heat, if too little of the solvent is not used. Upon mixing with a small, measured amount of water, a whole series of hydrates is formed; with hydrochloric acid, chlorostannic acid, H_2SnCl_6 , is obtained, crystallizing in plates which melt at 28° and contain 6 molecules of water. The ammonium salt, $(\text{NH}_4)_2\text{SnCl}_6$, is found in commerce under the name of *pink salt*. Tin tetrachloride forms crystalline compounds also with nitrous anhydride, with the chlorides of phosphorus and sulphur, as well as with hydrocyanic acid.

LEAD PEROXIDE, PbO_2 .

(a) *In a Dry Way.*

Literature: Kassner, *Chemische Industrie* **13**, 104, 120.

Mix 53 grams of lead oxide (see page 6) and 50 grams of calcium carbonate intimately in a mortar. Loosely pack a fire-clay crucible with this mixture, using a crucible which is not very deep but which is as wide as possible. Ignite in a Rössler gas-furnace for $\frac{1}{2}$ hour, being careful to have an excess of air in

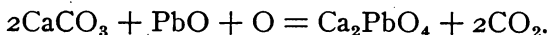
the space about the crucible by diminishing the amount of gas used, completely opening the air regulator, and using a large flame in the flue. After cooling, pulverize the frit (which has caked but slightly) in a mortar, and ignite again for $\frac{1}{2}$ hour at a bright red heat. Treat a pulverized sample of the product thus obtained with dilute nitric acid: if carbon dioxide is evolved, heat the pulverized mass again for a short time at a low white heat.* Gradually add the finely pulverized, flesh-colored calcium plumbate to 200 c.c. of dilute nitric acid; after settling, decant and grind up in a mortar with fresh nitric acid (100 c.c.). Finally, heat up with 100 c.c. of dilute nitric acid, filter, wash with hot water, and dry on the water-bath. For most purposes, it is more convenient to wash the peroxide by decantation with hot water and preserve as a paste. The weight of the lead peroxide obtained is about the same as that of the lead oxide used.

* The oxidation is accomplished very rapidly if the mixture is heated in a fire-clay crucible (iron is not suitable as it is strongly acted upon), in a Rössler gas-furnace, to the highest possible temperature. [Relative to the placing of the crucible, see sodium nitrite, page 6.] As soon as the mass begins to glow, stir thoroughly with an iron spatula, whereupon the mass, which at the beginning, in consequence of the easy fusibility of the lead oxide, is in a state of fusion, rapidly becomes completely dry and pulverulent. The small opening in the cover of the furnace is not closed during the operation. Make sure, by holding a splinter in front of it, that fresh air is continually coming in contact with the mixture to be oxidized.

(b) In a Wet Way.

Add a solution of 190 grams of lead acetate in 500 c.c. of water to 500 c.c. of a 20% sodium hydroxide solution, and treat the cooled, milky, alkaline liquid with chlorine, shaking during the operation, until no more precipitate forms. Decant with water and purify with nitric acid as above.

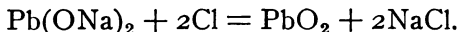
REACTION: In the presence of the oxygen of the air, lead oxide expels the carbon dioxide from the calcium carbonate, with the formation of calcium plumbate, which corresponds to the orthosilicates:



The plumbate, which varies but slightly from the color of the original mixture, is easily decomposed by nitric acid:



Lead peroxide is formed from the aqueous solution by the action of chlorine on the sodium plumbite:



TEST: Upon heating with dilute nitric acid, the dark-brown lead peroxide should impart no violet color to the supernatant liquid; this color should be formed, however, upon addition of a drop of dilute manganous sulphate solution.* Suspended in dilute

* A manganous salt is oxidized in nitric acid solution by lead peroxide to permanganic acid, and nitrous acid oxidized to nitric acid:



nitric acid, it should, upon the addition of sodium nitrite, go easily and completely into solution. To determine the amount of lead peroxide present, weigh off an average sample of the paste, or of the finely pulverized dried preparation; treat with dilute nitric acid in a flask having a ground-glass stopper, and gradually add normal sodium nitrite (page 7) in slight excess, aiding the decomposition by shaking the closed flask frequently and vigorously. After all the peroxide has gone into solution, dilute with much water and titrate back, with potassium permanganate, the excess of nitrite.

THORIUM DIOXIDE, ThO_2 .

Literature: Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 601.

It is most convenient to use the mantles which serve for incandescent lighting by means of gas, as a material for the preparation of pure thorium dioxide in the laboratory.

Grind to a paste 10 grams of the residues of such mantles (corresponding to about 20 mantles), with concentrated sulphuric acid; calcine to a dry powder, and add gradually, in a finely pulverized condition, to a mixture of 50 c.c. of water and 50 grams of ice, stirring very thoroughly during the addition. After any cerium present has been separated by means of

sodium sulphate, precipitate the thorium from the solution in the form of its hydroxide, $\text{Th}(\text{OH})_4$, by adding sodium nitrite. For further purification, the thorium hydroxide may be dissolved in citric acid, the impurities (iron, etc.) precipitated by means of ammonia and ammonium sulphide, and the thorium dioxide recovered by evaporation and ignition; for in the presence of organic acids, no single reagent will precipitate it.

REACTION: Thorium dioxide is insoluble in the ordinary solvents, but is converted into a very stable sulphate upon heating with sulphuric acid, which is only soluble in ice-water. Upon heating the aqueous solutions of the thorium salts, insoluble modifications are formed. Thorium dioxide forms no stable salt with nitrous acid.

TEST: Ignite 1 gram of thorium dioxide with sulphuric acid; dissolve the dry, finely pulverized sulphate in 10 c.c. of ice-water, and heat the filtrate: insoluble thorium sulphate separates, which, upon calcining, reforms the modification soluble in ice-water.—Precipitate the cold sulphate solution with ammonia and dissolve, in a small amount of nitric acid, the thorium hydroxide which separates. By evaporation at ordinary temperatures, the salt $\text{Th}(\text{NO}_3)_4 + 6\text{H}_2\text{O}$ crystallizes in pointed double pyramids.

NITROGEN TETROXIDE, N_2O_4 .

Gently warm 200 grams of commercial white arsenic (arsenious oxide), in pieces of the size of a pea, in a tubulated retort with 200 c.c. of nitric acid of a specific gravity of 1.38, the retort being fitted with a bent tube (as in Figure 4). Pass through an empty wash-bottle the reddish gas which is evolved; then dry it by means of a tube filled with calcium nitrate, and finally conduct it into a wide sealing-tube which is placed in a cooling mixture of salt and ice. A dark-green liquid condenses in this tube. Pass dry oxygen into this liquid until it is decolorized; then seal the tube.

REACTION: Arsenious oxide, As_2O_3 , reduces the nitric acid, being itself converted into arsenic acid.* In drying the gases, calcium chloride should not be used; for they would immediately contain chlorine. Neither should sulphuric acid be used, as it dissolves the gases. The condensed mixture of nitrogen trioxide and tetroxide is converted into pure tetroxide by oxygen.

TEST: The liquid (nitrogen tetroxide) should have a specific gravity of 1.45 and boil at $25-26^\circ$. By cooling strongly, it should solidify, forming colorless, prismatic crystals which melt at -9° .

* For the utilization of the arsenic acid residues, see page 68.

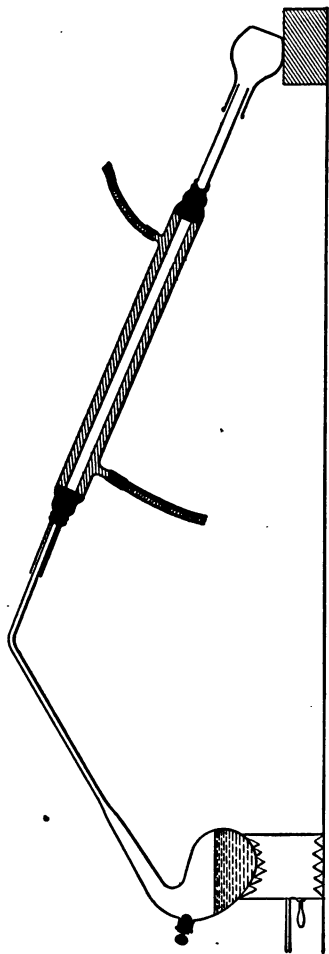


FIGURE 4.—Preparation of Pure Anhydrous Nitric Acid.

PURE ANHYDROUS NITRIC ACID, HNO_3 .

In a retort having a bent tube and an attached condenser (see illustration), distil on the gas-stove $\frac{1}{2}$ liter of commercial nitric acid with $\frac{1}{2}$ liter of concentrated sulphuric acid. Collect by itself the distillate which first comes over, until a sample caught in a test-tube no longer gives a precipitate with silver nitrate. At a gentle heat and in not too light a place, redistil, through the bent tube, the main portion mixed with an equal volume of pure concentrated sulphuric acid, testing the first portion of the distillate again for chlorine. Collect the anhydrous acid in a flask which is closed with glass wool. After the distillation is finished, place the flask, which is fitted with a double-bored rubber stopper, in a water-bath which is at 40° , and aspirate air through the acid by means of a water vacuum pump. The air must previously be dried by sulphuric acid and filtered through a tube filled with cotton. Keep the acid in a dark place.

REACTION: The water which is mixed with the nitric acid (an acid which begins to boil at 86°) is held back by careful distillation with sulphuric acid. Chlorine distils with the first portions. If no bent tube is used, sulphuric acid is constantly carried over mechanically into the distillate; also, by too rapid distillation, sulphuric acid vapors are carried over. Bright sunlight decomposes nitric acid. The oxides

of nitrogen can be removed from the acid by dry air. Absolutely pure and colorless nitric acid can be obtained by distillation under diminished pressure; * this operation, however, cannot be recommended for the purpose of practice; for it cannot be carried out safely except with special care in the method of procedure.

TEST: The acid should not show the slightest turbidity with silver nitrate. A few cubic centimeters evaporated on the water-bath, in a small platinum dish, should leave no weighable residue; and, upon washing out the dish with water, the liquid should not become turbid with barium chloride. (Direct treating of the nitric acid with barium nitrate, shows the presence of only larger amounts of sulphuric acid). The acid should be colorless and have a specific gravity of 1.54.

PURE AMMONIUM CHLORIDE, NH_4Cl .

Literature: Krüss, *Annalen* **238**, 51.

Dissolve 1 kilogram of commercial sal ammoniac in $1\frac{1}{4}$ liters of boiling water, filter through a hot-water filter, and heat with 100 c.c. of concentrated nitric acid until no more acid vapors are evolved. Filter off the ammonium chloride which separates upon cool-

* Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 175.

ing by means of a pump and recrystallize from hot water.

REACTION: The organic bases usually present in large amounts in commercial sal ammoniac (from gas-liquor) are destroyed by heating with nitric acid.

TEST: The salt should be bright white and dissolve clear in water, forming a transparent liquid. 5 grams volatilized in a weighed platinum dish at a gentle heat on the gas-stove, should leave no carbonaceous residue, and should always leave no weighable residue.

AMMONIUM DICARBONATE, $(\text{NH}_4)_2\text{HCO}_3$.

Place concentrated aqua ammonia in a small flask which is closed by a stopper bearing a short glass tube. Through this glass tube, which should extend nearly to the surface of the liquid but not dip into it, conduct carbon dioxide, generated in a Kipp apparatus. The neutral ammonium carbonate, which precipitates at first, redissolves, and, after the liquid has stood for some time under the pressure of the carbon dioxide, the acid salt separates. More dicarbonate is obtained from the mother-liquor by covering it with a layer of alcohol. Preserve the salt in glass tubes which have been filled with carbon dioxide and then sealed.

TEST: Ammonium dicarbonate forms hard, bright

rhombic prisms, which are very volatile. An aqueous solution should not become turbid upon the addition of calcium chloride.

HYDROCYANIC ACID, HCN.

(Prussic Acid.)

Literature: Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 463.

Distil on the gas-stove, in a well-ventilated place, 500 grams of coarsely pulverized yellow prussiate of potash, with 350 grams of concentrated sulphuric acid and 700 c.c. of water, in a retort having an ascending tube and a descending condenser (see illustration, page 58). Attach two Woulff bottles air-tight to the condenser, and surround the bottles with ice; conduct the vapors evolved from the last bottle into cold water. The insoluble residue formed in the retort * causes a little bumping; but, by an even, careful heating, the breaking of the retort need not be feared. The distillate is almost completely pure. If there is need for absolutely anhydrous hydrocyanic acid, fill the first Woulff bottle, previous to the distillation, half full of porous calcium chloride (page 24) and, at the end of the operation, place it in warm water, in order to distil its contents into the second bottle.—

* For the utilization of these residues, see prussian blue, page 112.

The product should not be kept in an anhydrous condition, but in dilute solution; and the aqueous hydrocyanic acid keeps still better upon the addition of a drop of dilute mineral acid to each 100 c.c. of the solution.

TEST: Anhydrous prussic acid is an extremely volatile, mobile, colorless liquid, which, when put upon the skin, produces a feeling of cold, like evaporating ether. It boils at 27° and solidifies to a crystalline mass in a cooling mixture. The aqueous solution does not redden litmus paper.

UREA, CON_2H_4 .

(1) Dissolve the crude potassium cyanate (which is prepared according to the method on page 10) from the frit, which contains chromic acid, by grinding in a mortar with cold water.* To the clear aqueous solution, add a concentrated solution of 150 grams of ammonium sulphate and evaporate to dryness on the water-bath, stirring during the evaporation. Extract the residue with alcohol, which upon evaporation yields urea. Crystallize the urea from amyl alcohol; † take up the residue from the evaporated mother-liquor in water, neutralize with nitric acid, and separate from

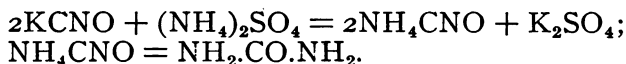
* Hot water decomposes the potassium cyanate (compare with page 12).

† 700-800 grams of amyl alcohol are necessary.

it the more difficultly soluble and easily crystallizable urea nitrate.

(2) After filtering off the cyanate which crystallizes out, immediately add the alcoholic mother-liquor (which contains potassium cyanate, mentioned on page 12) to a solution of 70 grams of ammonium sulphate in 100 c.c. of hot water, shaking during the addition. Distil off the alcohol, evaporate the residue to dryness with the addition of some barium carbonate, and extract with 96% alcohol, a considerable amount of the urea going into solution. By recrystallizing once from amyl alcohol, the carbamid is obtained quite pure.

REACTION:



TEST: Urea should form colorless, four-sided prisms or plates, having a melting-point of 132° , and should dissolve clear in an equal weight of cold water. The solution should give a crystalline precipitate with oxalic acid.

PHOSPHORIC ACID, H_3PO_4 .

Heat 127 grams of white phosphorus and 1400 c.c. of nitric acid (specific gravity 1.20) in a retort, having a receiver attached. The phosphorus melts and the reaction takes place quietly. From time to time,

the distillate must be poured back. When the phosphorus has gone completely into solution (after 10–12 hours), evaporate in a platinum dish, until a sample taken out with a glass rod no longer gives a test for nitric acid with concentrated sulphuric acid and ferrous sulphate solution. The temperature should not exceed 188° . The acid, treated with mercuric chloride solution, should show the absence of phosphorous acid. Then treat it, while warm, with hydrogen sulphide, until, even upon standing, no more sulphide of arsenic precipitates. Dilute with water, filter, and again slowly evaporate until a thermometer dipped into it shows a temperature of 160° .

TEST: The acid should show a specific gravity of 1.88 and be free from arsenic and phosphorous acid.

PHOSPHORUS TRISULPHIDE, P_2S_3 .

Mix 310 grams of red phosphorus with 480 grams of pulverized sulphur, and add, little by little, by means of a spoon, to a Hessian crucible which is heated with a Bunsen burner. After the addition of each portion, close the crucible with a cover, whereupon the reaction must immediately take place. After the whole of the mixture has been added, let the crucible cool to such an extent that the mass is just molten; then pour the sulphide of phosphorus upon an iron

plate. Break into pieces the solidified product, while it is still warm, and put it into a well-stoppered bottle.

Phosphorus trisulphide forms a hard, gray, easily pulverizable mass, which, upon standing in the air, becomes moist and slimy, hydrogen sulphide being evolved.

CALCIUM PHOSPHIDE, CaP .

Literature: Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 551.

Cut a round cover from strong sheet iron 2 mm. thick, to fit a crucible which is about $6\frac{1}{2}$ cm. wide and 12 cm. high, and which is made of dense, difficultly fusible clay. In the center of the cover have a circular opening 2.5 cm. in diameter, through which passes an iron tube 30 cm. in length. To the upper end of this iron tube, join a slightly smaller, thin-walled glass tube of 15 cm. in length.* After the iron tube has been placed upright in the crucible, fill the latter with 100 grams of lime (made from marble) in pieces the size of a hazelnut, put on the cover, and heat in the

* In order to accomplish this, wind some thin asbestos thread around the glass tube and twist it into the iron tube, having previously moistened the asbestos with water-glass. Cover the joints with a pasty mixture of powdered pyrolusite and water-glass, and dry at a gentle heat. The glass tube must be sufficiently wide to permit an easy passage of the pieces of stick phosphorus.

Rössler furnace (page 117). After the crucible has become red-hot, add, through the glass tube, 65 grams of stick-phosphorus in carefully dried pieces of 5-10 grams each. Immediately after the addition of each piece of phosphorus, close the glass tube with a cork.

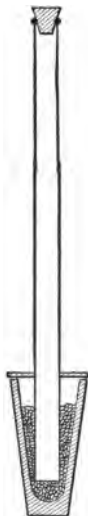


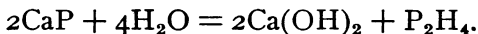
FIGURE 5.—Preparation of Calcium Phosphide.

The action of the phosphorus on the lime is made known each time by the evolution of phosphorus pentoxide in the form of white clouds from the chimney of the furnace. If the reaction fails to take place, lift up the iron tube a little, in order to aid the entrance of the phosphorus into the crucible. While at the beginning but a minimum of the phosphorus does not enter the reaction, the end of the operation is shown by the appearance of many fumes. Then ex-

tinguish the flame, lift the crucible out, and, as soon as the product has cooled down until it is but slightly warm, put it into a well-stoppered preparation bottle. Yield: 140 grams.

REACTION: $7\text{CaO} + 7\text{P} = \text{Ca}_2\text{P}_2\text{O}_7 + 5\text{CaP}$.

TEST: Calcium phosphide forms dark, hard, iridescent pieces, which, upon throwing into water, evolve spontaneously inflammable phosphoretted hydrogen:

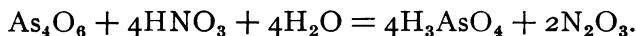


ARSENIC ACID, $2\text{H}_3\text{AsO}_4 + \text{H}_2\text{O}$.

Pour, from the unchanged pieces of white arsenic, the mother-liquor obtained in the preparation of nitrogen tetroxide (page 57), evaporate to dryness in a porcelain dish, and redissolve the residue, by continued heating, in a small amount of water. The solution must be free from arsenious acid; otherwise, it is again evaporated to dryness after the addition of nitric acid, and again redissolved. When the solution is pure, evaporate it to a syrup and let it crystallize in a closed flask at winter temperature, if necessary, with the addition of a small crystal of arsenic acid. In case just the right concentration is obtained, very beautiful large compact shining crystals form, which have the composition H_8AsO_9 ; and this compound probably corresponds to the constitution $(\text{HO})_4\text{As}-\text{O}-\text{As}(\text{OH})_4$. If crystallization results

too rapidly, filter off the mass; melt the crystals at a gentle heat in a flask having an air-tight stopper, or in a sealed tube, and allow the fused mass to crystallize slowly, obtaining larger crystals.

REACTION: The arsenious oxide is oxidized according to the equation:



Upon evaporating to dryness, as is necessary in order to get rid of the excess of the nitric acid, compounds are formed containing less water, which upon long treatment with a small amount of water go over into normal arsenic acid.

TEST: The acid should form transparent rhombic prisms or plates of the composition $2\text{H}_3\text{AsO}_4 + \text{H}_2\text{O}$, which are very deliquescent and dissolve in water, with a considerable lowering of the temperature. It melts at 100° , losing its crystal water and forming normal arsenic acid, AsO_4H_3 , which is also very easily soluble and crystallizes in small needles. The solution reacts and tastes strongly acid, and, after adding an excess of ammonia, gives with magnesia mixture a crystalline precipitate of magnesium ammonium arsenate, $\text{NH}_4\text{MgAsO}_4$, which upon being treated with silver nitrate solution changes into reddish-brown silver arsenate, Ag_3AsO_4 .

The solution of arsenic acid should give no test for nitric acid with ferrous sulphate. A highly diluted solution treated with a drop of hydrochloric acid

should, upon adding an equal volume of hydrogen sulphide solution, remain at first clear, and should become turbid only after long standing. A solution of the acid in fuming hydrochloric acid should give immediately, with hydrogen sulphide gas, a thick yellow precipitate of arsenic pentasulphide, As_2S_5 , which must be filtered off and washed without warming. Hydrogen sulphide does not act on a cold, weak acid solution of arsenic acid, but, under such conditions, pure arsenic pentasulphide, As_2S_5 , precipitates from a warm solution. Arsenic pentasulphide is of great importance in the analytical separation and determination of arsenic. It is the most convenient form in which arsenic can be obtained for weighing.* [Concerning the treatment of arsenic compounds for the presence of antimony, and the separation of arsenic from antimony, see Erdmann, *Lehrbuch der anorganischen Chemie*, pages 394 and 403.]

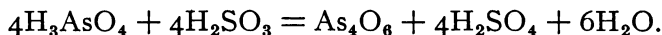
* These conditions have been already established by Robert Bunsen, but the directions of Bunsen have recently been assailed in an unjustifiable manner. Piloty and Stock first showed, in 1897, that Bunsen's method for precipitating arsenic as pentasulphide is, in fact, an excellent one, if the precipitation is carried out exactly according to Bunsen's directions, in a warm, very weak hydrochloric acid solution; for warm concentrated hydrochloric acid partially reduces the arsenic acid, and even changes the sulphide already precipitated.

CRYSTALLINE ARSENIOS OXIDE, As_4O_6 .*(Flowers of Arsenic, White Arsenic.)*

Literature: Friedheim and Michaelis, *Berichte d. d. chem. Ges.* **28**, 1417, note; Erdmann, *Lehrbuch der anorganischen Chemie*, page 385.

(1) With a freshly prepared saturated aqueous solution of sulphurous acid (page 16), dilute to 1 liter 75 grams of the syrup which remains after the preparation of crystalline arsenic acid (page 68), and let it stand in a well-stoppered flask. During the course of a few weeks arsenious oxide separates in beautiful shining octahedra. Pour off the liquid, and wash the crystals with a little water, alcohol, and ether.

REACTION:



(2) Dissolve commercial arsenious acid to a syrup in 50% caustic potash solution, and dilute with twenty times its volume of water. At first, crusts which are indistinctly crystalline separate; but if the solution is poured off from these, larger individual octahedra are obtained.

REACTION: The salts of arsenious acid are stable only in the presence of an excess of alkali, or in concentrated solution; but when much water is present, they decompose, with the formation of arsenious oxide.

(3) Treat 40 grams of commercial arsenious acid, in

a 250 c.c. distilling-flask, with 100 c.c. of methyl alcohol; then pass in hydrochloric acid, cooling down the liquid as it warms up, in order to avoid a premature loss of the alcohol. After saturation, distil on the water-bath into a well-closed receiver, stopping the distillation when 5 c.c. of liquid still remains in the flask; then dilute the distillate with water, until crystals just begin to separate. Filter off the crystalline arsenious oxide, wash with water, and dry at 110° .

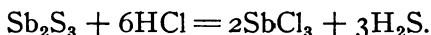
REACTION: The arsenic is converted into the quite volatile methyl ester of arsenious acid, while the main portion of the antimony, which is there as an impurity, remains behind. The ester is decomposed by water; and arsenious oxide separates, while any antimony that has passed over remains in solution in the very strongly acid liquid.

TEST: Heat a small crystal in a dry test-tube or on a piece of platinum foil: it should volatilize, leaving no residue (though with the octahedra prepared according to the second method this is not always the case, because they frequently contain some alkali).—Weigh 0.4912 gram of the pulverized arsenious oxide into a large beaker, add 100 c.c. of water and 1 gram of sodium dicarbonate, and titrate with normal iodine solution. 10 c.c. of the normal solution should be required.

ANTIMONIOUS CHLORIDE, SbCl_3 .

In a flask under a hood, heat 100 grams of powdered stibnite with 500 c.c. of commercial hydrochloric acid, adding gradually about 4 grams of potassium chlorate. When the ore has disappeared, filter off the sulphur through glass wool, and distil in a retort. At first aqueous hydrochloric acid comes over, then a concentrated antimony trichloride solution colored yellow by ferric chloride, and finally pure antimony trichloride, which solidifies to a beautiful white crystalline mass. Each of these products is collected separately. The pure antimony trichloride is sealed in a preparation-tube or a small flask; and the antimony trichloride solution is decomposed with much cold water, thus precipitating antimony oxychloride as a fine white powder.

REACTION: The reaction, which at first is energetic, becomes slower, but is hastened by the addition of the oxidizing agent.



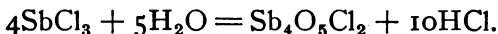
TEST: Antimony trichloride should melt at 73° , boil at 223° , and crystallize from carbon disulphide in bright rhombic crystals.

BASIC CHLORIDE OF ANTIMONY, $\text{Sb}_4\text{O}_5\text{Cl}_2$.*(Powder of Algaroth.)*

Literature: G. E. Stahl, Zufällige Gedanken und nützliche Bedenken über den Streit von dem sogenannten Sulfure, Halle 1715, page 346.

Intimately mix 60 grams of pulverized sulphide of antimony and 140 grams of mercuric chloride by means of a mortar, and distil slowly from a glass retort on the gas-stove, using a small flame. For a receiver, use a flask, the neck of which has been broken off, and heat the neck of the retort from time to time with a Bunsen burner, in order to melt the antimony trichloride which has solidified in it. Dissolve the distillate in a small amount of warm hydrochloric acid, and pour it into a large amount of hot water. Wash the precipitate which forms, by decantation, and dry it.

REACTION:



TEST: The white, crystalline powder should be insoluble in water, alcohol, and ether, but soluble in carbon disulphide and in chloroform. Upon heating, it should give a sublimate of antimonious chloride and leave a residue of oxide of antimony.

SULPHATE OF ANTIMONY, $\text{Sb}_2(\text{SO}_4)_3$.

Add 20 grams of finely pulverized antimony to 400 grams of pure boiling sulphuric acid, and heat in a platinum dish until the metal has disappeared. A portion of the sulphate crystallizes in small white needles, while the acid is still hot; the remainder crystallizes upon cooling. Filter on a platinum cone, and dry the preparation on a porous plate.

BISMUTH NITRATE, $\text{Bi}(\text{NO}_3)_3 + 5\text{H}_2\text{O}$;

BASIC BISMUTH NITRATE,
 $\text{BiO}.\text{NO}_3 + \text{BiO}.\text{OH}$;

BISMUTH HYDROXIDE, $\text{BiO}.\text{OH}$.

Heat to a low red heat 100 grams of commercial bismuth with 50 grams of sodium nitrate, using a nickel dish. After the metal is completely oxidized, heat with 250 c.c. of water, with the addition of a solution of 20 grams of sodium hydroxide in 150 c.c. of water. After filtering off the bismuth oxide on a platinum cone, dissolve the oxide in a hot mixture of 140 c.c. of concentrated nitric acid and 200 c.c. of water; filter through asbestos, and evaporate to crystallization.—Evaporate the mother-liquor considerably and pour it into $\frac{1}{2}$ liter of boiling water, the basic bismuth nitrate separating as a heavy white powder.

After decanting, collect this on a filter and dry without warming.—Or, precipitate the acid liquor with ammonia, wash the bismuth oxyhydrate by decantation, and preserve as a paste.

REACTION: Bismuth, such as is used in the arts, has usually arsenic and antimony as impurities, the oxides of which dissolve in sodium hydroxide upon heating.

TEST: The amount of bismuth in the crystallized nitrate is determined in a weighed sample, by converting it into the oxide by first carefully heating in a covered porcelain crucible, then heating strongly and weighing the product. Also dissolve a sample in water acidified with nitric acid, add this solution to an excess of hot sodium hydroxide, and test the filtrate for arsenic and antimony.

BISMUTH IODIDE, BiI_3 .

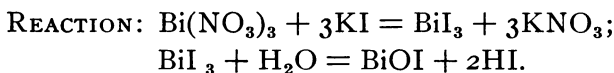
Grind 20 grams of iodine with 35 grams of finely pulverized bismuth in a mortar, introduce rapidly into a retort, and heat slowly on the gas-stove. After union has taken place, drive off the small excess of iodine by means of a current of carbon dioxide; then raise the temperature until the bismuth iodide sublimes in very large crystals, which resemble iodine.

TEST: Boiling water should convert the powdered preparation into the red oxyiodide.

BISMUTH OXYIODIDE, BiOI.

Literature: B. Fischer, *Die neueren Arzneimittel*, III. Aufl. page 20.

Dissolve 95.4 grams of the pulverized crystalline bismuth nitrate (page 75) by gently heating with 120–160 c.c. of glacial acetic acid; also dissolve 33.2 grams of potassium iodide and 50 grams of crystallized sodium acetate in 2 liters of cold water. Place the second solution in a dish and let the first flow into it in a very small stream from a drop-funnel, stirring constantly during the addition. At the place where the liquids meet, a greenish-black precipitate forms at first, which, upon stirring, changes to a lemon-yellow color. Upon further addition of the bismuth nitrate solution, the product changes to a brick-red color. The precipitate settles very well. Decant, filter, and dry at 100°. Yield: 78 grams.



The hydriodic acid is decomposed by the sodium acetate, and the alkaline iodide thus formed again enters the reaction.

TEST: Heated in a dry test-tube, the preparation should give off violet iodine vapors, and leave a residue of bismuth oxide. Upon shaking with water, it should produce no haloid acid, and should be free from arsenic (test according to directions on page 27,

with the addition of zinc) and subnitrate of bismuth. —0.2 gram shaken with 2 grams of dilute sulphuric acid should give a filtrate which, when mixed with twice its volume of concentrated sulphuric acid, should produce, upon adding a drop of indigo solution, a permanent blue color (decolorization indicates nitric acid).

HYDROGEN PEROXIDE, H_2O_2 .

Literature: Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 154.

(1) Mix 20 c.c. of concentrated sulphuric acid with 200 c.c. of water in a beaker, carefully cool the liquid in a cooling mixture of ice and salt, and add, while stirring, moist hydrated barium peroxide, until the solution has but a slight acid reaction. Let the solution settle, filter, and add to the liquid, which is still ice-cold, dilute barium hydroxide, until a test by the capillary method * shows the presence of neither sulphuric acid nor barium in the solution. Filter the turbid liquid and distil in vacuo, at about 20 mm. pressure.

(2) It is more convenient to start with a 3% solution of commercial hydrogen peroxide. For this purpose, do not take less than $\frac{1}{2}$ liter of hydrogen peroxide solution; place it in a thick-walled, round

* See note, page 2.

bottomed 1-liter flask having a long neck, and evaporate to about 100 c.c. on the water-bath, at 75°, the flask being connected with a filter-pump. Distil the residue from a fractioning-flask, in the most complete vacuum obtainable,* catching the distillate in a receiver cooled by ice. At first, aqueous hydrogen peroxide distils; finally, the pure product, the impurities remaining behind.

REACTION: The reaction,



takes place only in a cold solution, and at not too great a concentration. If these conditions are not adhered to, much oxygen is evolved, and the yield is reduced to a minimum.

TEST: Anhydrous hydrogen peroxide should form a thick syrup, which immediately blisters the skin white; it should decompose easily, should have a specific gravity of 1.46 and a boiling-point of 69° at 20 mm. pressure. The aqueous solution should have a peculiar characteristic taste, and should give with dilute sulphuric acid, ether, and a drop of dilute potassium dichromate solution, an intensely blue-colored compound, soluble in ether. It should be free from hydrochloric acid, sulphuric acid, and barium. For preserving, it is made more stable by the addition of 1% alcohol. To determine the amount of hydrogen

* To render a cork stopper air-tight in a vacuum distillation, use thick collodion, laid on by means of a brush or a feather.

peroxide present, decompose 1 c.c. of the solution, in a nitrometer, with an excess of permanganate and dilute sulphuric acid; or, titrate 5 c.c., in sulphuric acid solution, with tenth normal permanganate.

DISULPHUR DICHLORIDE, S_2Cl_2 ;
SULPHUR DICHLORIDE, SCl_2 .

Melt 300 grams of sulphur in a tubulated retort on the gas-stove, using a small flame, and conduct through a glass tube a very rapid current of dry chlorine close to the surface of the melted sulphur. Rectify, from a flask, the chloride (which distils rapidly), using a fractioning-column; and there is left a residue of the sulphur which was dissolved.

Cool 67.5 grams of the disulphur dichloride thus obtained, to 0° , and saturate it with dry chlorine. The sulphur dichloride obtained in this manner must weigh 103 grams. Preserve this product in a bottle that is well-stoppered and wired.

REACTION: Upon rectifying the chloride of sulphur, the higher chlorides change to disulphur dichloride; on the other hand, at 0° , disulphur dichloride unites with another molecule of chlorine:



TEST: Disulphur dichloride should form a yellowish-red, strong-smelling liquid, of a specific gravity of

1.7, and should have a boiling-point of 138° . The sulphur dichloride should form a dark-red liquid, which evolves chlorine even at ordinary temperatures, but more rapidly upon heating.

BARIUM DITHIONATE, $\text{BaS}_2\text{O}_6 + 2\text{H}_2\text{O}$;
DITHIONIC ACID, $\text{H}_2\text{S}_2\text{O}_6 + x\text{H}_2\text{O}$.

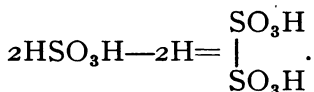
Levigate 100 grams of finely pulverized pyrolusite by stirring it in a large mortar with water, decant, and grind the residual coarser powder with more water. Continue the grinding and levigation until all the mineral remains suspended in water; let it settle in a tall cylinder, siphon off the water, and add only $\frac{1}{2}$ liter of water to the pyrolusite in a liter flask.* Pass a current of sulphur dioxide into the flask, and, by cooling well on the outside with ice, prevent a considerable rise in temperature of the contents of the flask. When the principal portion of the pyrolusite has gone into solution, add to the heated liquid concentrated barium hydroxide in a dish, until a filtered test no longer gives a flesh-colored precipitate with ammonium sulphide. Filter the precipitate, extract it with hot water, unite the filtrates, and treat, while hot, with carbon dioxide until the alkaline reaction disappears.

* Precipitated manganese dioxide may be used immediately for this experiment without any further treatment.

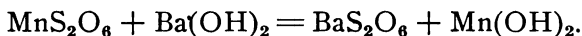
Barium dithionate crystallizes from the filtrate after concentration.

Dissolve 59 grams of the barium dithionate in water. Dilute 20 grams of sulphuric acid with water, and add such an amount of it to the barium salt that a test, withdrawn by means of a capillary tube,* reacts neither with sulphuric acid nor with barium chloride. After filtering off the barium sulphate, concentrate the dithionic acid in a shallow porcelain plate until a specific gravity of 1.347 is reached, the concentration being carried on in vacuo over sulphuric acid.

REACTION: The peroxide takes up two atoms of hydrogen from two molecules of sulphurous acid:



The manganese salt of dithionic acid is decomposed by the excess of barium hydroxide:



TEST: Barium dithionate should form beautiful, bright, colorless crystals, which, in a powdered condition, lose all their crystal water (10.81%) at 100°. Determine the amount of barium (45.96% BaO) by igniting the dehydrated salt and weighing the residual barium sulphate.

* See note, page 2.

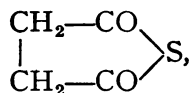
THIOPHENE, C_4H_4S .

Literature: Erdmann, *Präparatenkunde* (Stuttgart 1894), Bd. II, 548.

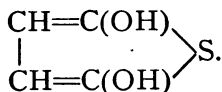
Dissolve 572 grams of crystallized sodium carbonate in 500 c.c. of water in a large porcelain dish, and, while stirring, gradually add 236 grams of succinic acid to the warm alkaline liquid. After boiling off the carbon dioxide, evaporate to dryness the liquid, which now reacts neutral, and finely pulverize the very hard mass of sodium succinate. In order not to be troubled in this operation by the dust, which strongly attacks the mucous membranes, moisten the salt with a few drops of alcohol. Dry the succinate very carefully at 140° . Intimately mix 300 grams of this fine powder, which no longer cakes together, with 400 grams of phosphorus trisulphide (page 65); place the mixture in a retort, and heat with a small flame on a gas-stove. As soon as any portion of the mass becomes dark, and gas begins to be evolved, reduce the size of the flame until it is barely visible, in order that the reaction may not become too violent. Notwithstanding this, the reaction, when once started, continues vigorously; and special precautions must be taken to condense the thiophene vapors, which are mixed with much gas, in order to obtain a satisfactory yield (40–50% of the theoretical yield). For this purpose, it is most suitable to attach directly to the retort, a glass tube, 2 cm. in diameter and 1–2 meters long,

which acts as an air-condenser. This tube is joined to a Liebig condenser, which is as long as possible and to which is attached an air-tight receiver. Pass the gases escaping from the receiver into an absorption-flask containing sodium hydroxide, such as is described on page 124. Rectify the distillate on a water-bath, once over powdered potassium hydroxide, and again over metallic sodium which is cut into fine pieces.

REACTION: The sulphide of phosphorus converts the sodium succinate into sulphosuccinyl,



which rearranges itself, forming dioxythiophene,



The latter is reduced to thiophene by the further action of the sulphide of phosphorus.

TEST: Thiophene, $\text{C}_4\text{H}_4\text{S}$, should form a very mobile, limpid liquid, boiling at $83-84^\circ$. The odor should remind one of benzene, and, at the same time, of sulphur compounds, but should not be disagreeable. In a cooling mixture of solid carbon dioxide and ether, it should solidify to a crystalline mass.—Dissolve a small grain of isatin in concentrated sulphuric acid which is not completely anhydrous; add

a drop of thiophene to 10 c.c. of pure benzene or petroleum ether, and shake 1 c.c. of each solution together. The sulphuric acid should not become dark-brown in color, but should turn greenish at first, and then should change rapidly into a splendid dark-blue color.

ANHYDROUS CHROMIC CHLORIDE, CrCl_3 .

Form a paste by kneading together the chromic oxide obtained according to the directions on page

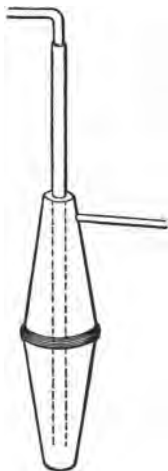


FIGURE 6.—Preparation of Anhydrous Chromic Chloride.

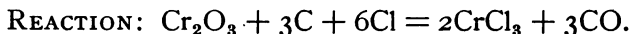
87, while it is still moist, with 50 grams of powdered charcoal and thick starch paste. Make rods of it, cutting them in pieces about 2-3 cm. long. Pack the

pieces, which have been dried at a gentle heat with powdered charcoal, in a Hessian crucible, covering all with a layer of charcoal. Close the crucible well with an iron cover (see page 27), and ignite in a Rössler furnace (page 117) for a quarter of an hour. Place a porcelain tube upright in a Hessian crucible, and fill the crucible with the pieces of chromic oxide and carbon, after they have become cooled and have been separated from the charcoal powder. Place in an inverted position on this crucible another one of the same size,* having an opening which the porcelain tube fits. A short glass tube passes through a second opening at the side of the upper crucible. Wind asbestos string about the tubes, thus rendering them air-tight at the points where they pass through the openings. In order to join both the crucibles tightly, place a thin narrow strip of soft asbestos paper about the place where the edges meet, and carefully wind asbestos thread around it. Finally, saturate the asbestos thread with water-glass, and dry it at a gentle heat. Place the crucible in a Rössler furnace and cover it with two semicircular pieces of thick sheet iron, cut out in the centre, through which the upper crucible protrudes from the furnace. Heat in a current of carbon dioxide, until no trace of moisture deposits in the glass tube. Increase the temperature

* The fire-clay crucibles are easily perforated by the metal drills which are to be found in any mechanic's workshop.

to the greatest extent and ignite in a current of chlorine, absorbing, in an absorption-bottle (page 124) containing sodium hydroxide, the unused chlorine which issues from the glass tube.

After cooling, the chromic chloride is found sublimed in the upper crucible in the form of very bright violet-red plates, which are insoluble in water.



CHROMIC OXIDE, Cr_2O_3 .

Literature: H. Schäffer, *Bull. de Mulhouse* 1893, 97; P. Werner, *ibid.* page 98; Erdmann, *Lehrbuch der anorganischen Chemie*, page 641.

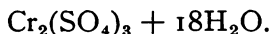
Treat 40 c.c. of glycerine, of a specific gravity of 1.23, at a moderate temperature, in a large nickel dish or copper kettle of 10 liters capacity, with 250 grams of sodium dichromate to which 15 c.c. of water has been added. Stir thoroughly, and warm at the same time on a water-bath. The homogeneous brown syrup which is formed soon catches fire spontaneously, upon stirring vigorously. If this is not the case, ignite it with a taper and cover the kettle immediately with a suitable sheet-iron cover which is ready for that purpose. Upon cooling, the kettle is found loosely filled with green chromic oxide. Wash it with cold water, then with warm water, filter on a Büchner funnel, dry on the water-bath; and ignite in

a closed crucible the oxide thus purified. The yield amounts to about 100 grams.

REACTION: The reduction of the dichromate by the glycerine takes place in such a way that, besides the chromic oxide formed, sodium carbonate is formed also. This, together with the small residue of unchanged chromate, is dissolved out with water. Any remaining traces of organic matter are removed by the ignition.

TEST: Upon heating the beautiful green powder with dilute hydrochloric acid, it should not go into solution, or only in traces.

VIOLET CHROMIUM SULPHATE,



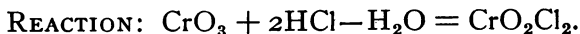
Literature: Etard, *Compt. rend.* **84**, 1089.

Dissolve 100 grams of chromic acid in 125 c.c. of water, and add to it a cooled mixture of 150 grams of concentrated sulphuric acid and 100 c.c. of water. Place this chromic acid solution in a porcelain dish, arrange on it a tripod carrying a porcelain crucible that contains 50 c.c. of ether, and cover the whole with a bell jar. When the ether has evaporated (and evaporation takes place pretty rapidly), it must be renewed. The chromium solution is completely reduced in a few days and converted into a magma of small, thin, violet scales, to which the thick green

chromium solution adheres and from which it cannot be freed by filtration. Consequently, it is best to dissolve the whole mass in the smallest possible quantity of cold water and precipitate with alcohol.

CHROMYL CHLORIDE, CrO_2Cl_2 .

Melt, in a Hessian crucible, 200 grams of neutral potassium chromate with 122 grams of salt, at not too high a temperature, and pour the molten mass upon an iron plate. Treat the coarsely powdered pieces, little by little, in a large retort, having a condenser attached, with a mixture of 66 c.c. of fuming sulphuric acid (specific gravity 1.906) and 134 c.c. of ordinary concentrated sulphuric acid. When the reaction, which at first is very violent, has moderated, heat until no more brown drops come over, and rectify the distillate from a small fractioning-flask. Preserve the chromyl chloride in a sealed flask.



TEST: The deep red oxychloride, which fumes in the air, should boil at 116° .

CHROMOUS ACETATE, $(\text{CH}_3\text{COO})_2\text{Cr}$.

Literature: v. d. Pfordten, *Annalen* **228**, 113; Erdmann, *Lehrbuch der anorganischen Chemie* (Braunschweig 1898), page 645.

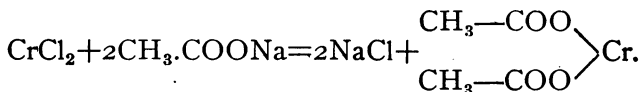
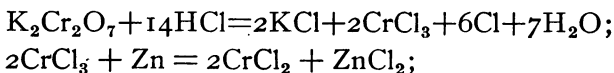
Treat 100 grams of potassium dichromate with 500 grams of fuming hydrochloric acid, in a flask; and, after washing with water, utilize the chlorine that is evolved in a steady current upon heating, to prepare chlorine water or sodium hypochlorite.* Evaporate the rest of the liquid to a very small volume on the gas-stove; decant from the potassium chloride which has separated, into a flask containing 300 grams of granulated zinc; and afterwards rinse the flask out, with 400 c.c. of fuming hydrochloric acid. Close the flask, in which a very violent evolution of hydrogen takes place, with a two-hole rubber stopper, provided with glass tubes similar to the top of a wash-bottle. As soon as the liquid becomes a clear bright blue color, which reminds one of a solution of copper sulphate, close the glass tube that affords the gas a free exit, so that the hydrogen, which is still vigorously evolved, forces the liquid out of the flask through the second tube, which reaches to the bottom of the flask.† The liquid passes, for filtration, to a small

* It may also be used in the preparation of potassium chlorate (page 13), silicon tetrachloride (page 48), stannic chloride (page 50), disulphur dichloride (page 80), chromic chloride (page 85), or ferric chloride (page 109).

† If the evolution of hydrogen is no longer sufficiently strong

bulb-tube containing asbestos, and then direct, without coming in contact with the air, into a solution of 500 grams of crystallized sodium acetate in 2 liters of water. Wash the red precipitate a number of times by decantation with water through which carbon dioxide has been passed, and preserve as a paste.

REACTION:



The reduction of the chromic chloride takes place smoothly and quickly only in a concentrated and very strong hydrochloric acid solution, with a great excess of zinc. Chromous chloride absorbs oxygen from the air with great rapidity, while the insoluble chromous acetate is tolerably stable in the air.

TEST: The red paste should give, with dilute hydrochloric acid, a blue solution, which rapidly turns dark-green with energetic absorption of oxygen.



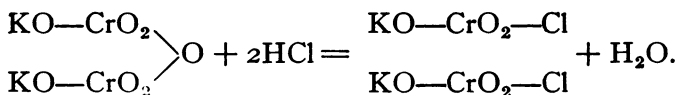
Pulverize 100 grams of potassium dichromate, and gently warm it in a flask with a mixture of 100 c.c. of

for this purpose, pass in carbon dioxide, and by this means force the chromous chloride solution over.



water and 130 grams of pure fuming hydrochloric acid. As soon as solution has taken place, filter, and let it stand quietly. On the following day, drain the mother-liquor from the crystals which have separated, and dry them on a porous plate. Yield: 65 grams.

REACTION:

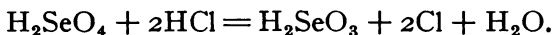


TEST: The large red prisms or plates evolve chlorine upon heating to 100°.

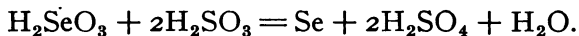
SELENIUM, Se.

Melt, in a Hessian crucible, any material or residues containing selenium, with a mixture of equal parts of sodium carbonate and potassium nitrate. Extract the cooled mass with hot water, evaporate the extract to a small volume, and by means of a return condenser heat the liquid, which is made strongly acid with concentrated hydrochloric acid, until no more chlorine is evolved. Dilute in a large flask with a large quantity of hot water, and drop in sodium disulphite, such as is used in the arts (40% solution), into the boiling liquid, just as long as a red, voluminous precipitate forms, which very rapidly mats together into an insignificant black mass. After settling, collect on a filter the selenium which has separated and dry it.

REACTION: Upon melting with sodium carbonate and potassium nitrate, material containing selenium forms selenates; but the selenic acid is very rapidly reduced in a concentrated solution of hydrochloric acid:



The selenious acid is decomposed by the sulphurous acid:



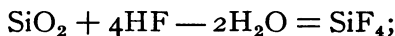
TEST: The selenium should melt at 217° , and solidify, upon cooling slowly, to a dark-gray crystalline mass of metallic lustre. It should dissolve in concentrated sulphuric acid, with a green color, and, upon the addition of water, precipitate again from this solution as a voluminous red amorphous precipitate. Upon heating, it should burn in the air, producing a radish-like odor, leaving no residue, and subliming to selenium dioxide.

FLUOSILICIC ACID, H_2SiF_6 .

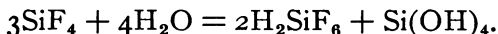
Mix 100 grams of powdered fluorspar with 100 grams of dry quartz sand (sea sand), and carefully heat in a flask with 350 c.c. of concentrated sulphuric acid. Conduct the gas evolved through an empty bottle fitted with a safety-tube, which is closed by

means of a small amount of concentrated sulphuric acid; and then conduct it into a porcelain dish, on the bottom of which is placed a very small bottle containing mercury. Fasten the delivery-tube in such a manner that it dips under the mercury, and add 400 c.c. of distilled water. After the gas evolution has ceased, drain the silicic acid which has separated in the water, press it out, wash with a little water until the combined filtrates amount to 400 c.c., and filter the still somewhat turbid acid through a creased filter.

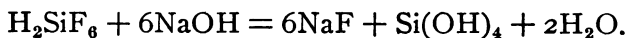
REACTION: Under the dehydrating influence of the sulphuric acid, silicon tetrafluoride is formed:



but this gas is decomposed by water:



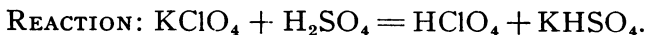
TEST: The acid should give a precipitate with barium chloride, but not with strontium chloride in a hydrochloric acid solution (sulphuric acid). To determine the amount present, titrate the acid with sodium hydrate to an alkaline reaction. The decomposition of the acid, upon titrating with sodium hydroxide, corresponds to the equation:



PERCHLORIC ACID, HClO_4 .

Literature: Caspari, *Zeitschr. angew. Chem.* 1893, 68; Erdmann, *Lehrbuch der anorganischen Chemie*, page 310.

(1) Distil, from a retort not too small, 50 grams of potassium perchlorate with a mixture of 100 grams of concentrated sulphuric acid and 20 c.c. of water. Free the distillate from any chlorine dissolved in it, by gently warming, treat with a few centigrams of silver sulphate and barium carbonate, filter the small precipitates separately, wash with a small amount of water, and again distil the filtrate.



The impure acid still contains traces of hydrochloric acid and sulphuric acid, which are removed by the method given.

(2) Dissolve 56 grams of potassium perchlorate in 400 c.c. of hot water and add to the hot solution, while stirring, 70 c.c. of fluosilicic acid of a specific gravity of 1.35 (31% H_2SiF_6). Heat for an hour with a return condenser; cool, decant, and filter off the potassium fluosilicate on a Büchner funnel. On a water-bath evaporate the clear solution of perchloric acid, as much as possible, and again filter, but through asbestos. To obtain the filtrate completely free from fluosilicic acid, treat the cold solution, after diluting with an equal volume of water, with a solution of about 2 grams of barium chloride, and evaporate the

liquid, which settles clear after a few hours, until the odor of hydrochloric acid has disappeared and white vapors of perchloric acid begin to be evolved.

TEST: A drop of the acid should produce, in a solution of potassium chloride, a thick crystalline precipitate.

HYDROBROMIC ACID, HBr .

For the preparation of hydrobromic acid gas, use the apparatus illustrated in Figure 7. Place in the

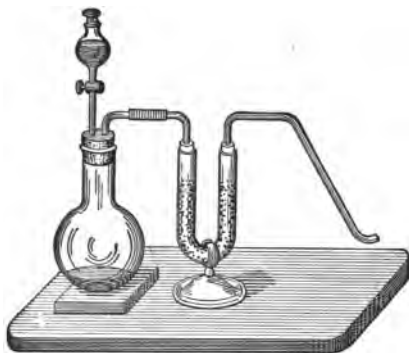


FIGURE 7.—Preparation of Hydrobromic Acid.

flask 100 grams of dry benzene and a few grams of anhydrous ferrous bromide (very fine iron powder or aluminium powder may be used in place of ferrous bromide). Gradually run in 135 c.c. of bromine from a separating-funnel, the end of which is drawn out to a fine point. The mixture immediately becomes

warm, and must be placed in cold water to prevent benzene or bromine from distilling over; but when half of the bromine has been added (formation of monobrombenzene), the reaction runs so quietly that this precaution is no longer necessary. In order completely to free the gas, which is evolved in a very steady current, from the benzene and bromine vapors which are carried over, use a U tube attached to the flask (Figure 7). Fill the first half of the tube with ferric bromide, FeBr_3 ,* the second half with anthracene, a solid hydrocarbon. For the preparation of a concentrated aqueous hydrobromic acid, conduct the gas into a small flask, add to it, little by little, small amounts of water from a wash bottle, and cool with a cooling mixture of snow and salt. Preserve the saturated liquid in a bottle having a well-ground stopper and glass cap, or, wire the stopper. Preserve in a dark place.

REACTION: $\text{C}_6\text{H}_6 + 4\text{Br} = \text{C}_6\text{H}_4\text{Br}_2 + 2\text{HBr}$. The small amount of ferrous bromide acts as a bromine carrier. The hydrobromic acid which is evolved in a very steady current, carries benzene vapors over; and as these vapors are brominated in the tube containing the ferric bromide, they consequently are retained. The gas is freed from the smallest traces of free bromine by means of the anthracene.

* Prepared by mixing 25 grams of ferrous bromide and 3 c.c. of bromine, keeping cold.

TEST: The aqueous solution should be completely colorless, should fume strongly in the air, and should have a specific gravity of at least 1.78 (the acid saturated at 0°).

AMMONIUM BROMIDE, NH_4Br ;
POTASSIUM BROMIDE, KBr .

Run 75 c.c. of bromine slowly from a drop-funnel, the stem of which has been drawn out to a fine point, into 220 c.c. of concentrated (30%) ammonia placed in a flask surrounded by ice-water, with the precaution that the liquid remains strongly ammoniacal till the end of the reaction, shaking continually during the addition. Then heat until the free ammonia has been driven off, and evaporate to crystallization. Dry the ammonium bromide by heating gently in a porcelain dish over a free flame. Yield: 220 grams.

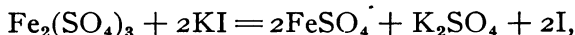
Dissolve 196 grams of ammonium bromide in hot water and add 200 grams of potassium dicarbonate; heat to boiling, and, after the odor of the ammonia has disappeared, set aside to crystallize.

REACTION: $4\text{NH}_3 + 3\text{Br} = 3\text{NH}_4\text{Br} + \text{N}$. If the solution becomes acid, or the bromine runs in too rapidly, bromide of nitrogen may be formed and may give rise to an explosion.

TEST: Heat a small sample of the salts in a small retort, with a large excess of iron ammonium alum

(page 111) in aqueous solution, whereupon any iodine present passes over in a free condition. Drive out the bromine in the residue, by the addition of a solution of potassium permanganate, and, after the removal of the bromine, test for chlorine by heating with alcohol the liquid which contains manganese peroxide and permanganate; then add yellow fuming nitric acid until the peroxide dissolves, and test with a drop of silver nitrate solution.

This separation of the halogens rests chiefly upon the fact that ferric salts set iodine free from neutral solutions of iodides,



while the bromides remain completely unattacked. On the other hand, even these are completely decomposed by ferric salts, if a small amount of a strong oxidizing agent (permanganate) is present. The chlorides withstand completely the simultaneous action of both these agents.

DIBROMBENZENE, $\text{C}_6\text{H}_4\text{Br}_2$.

The residue from the preparation of hydrobromic acid (page 96) solidifies to a crystalline mass. Melt it by placing the flask in warm water, and gradually bring the water to boiling. At the same time, free the substance as much as possible from the hydrobromic

acid present, pour into a retort, and distil. The distillate solidifies to a crystalline mass, which should be pressed out and again distilled, whereupon the main portion passes over at $215-220^{\circ}$. Recrystallize this last distillate from the same weight of hot alcohol. Yield: about 110 grams of pure dibrombenzene.

REACTION: Together with the dibrombenzene, there are formed some liquid monobrombenzene and *o*-dibrombenzene, which are removed by the pressing, while the higher brominated products remain in the residue from the distillation.

TEST: Dibrombenzene forms white monoclinic prisms or plates. It should melt at $87-88^{\circ}$ and boil at 219° .

POTASSIUM IODIDE, KI.

Suspend 6 grams of pure iron powder in 50 c.c. of water in a small flask, and gradually add 25 grams of iodine, keeping the liquid cool. At the end of the reaction, there must still be some excess of iron present. Filter it off, wash with a little water, and add 5 grams of iodine to the filtrate. Dissolve 16.5 grams of potassium carbonate in 50 grams of hot water in a porcelain dish, and add the iodide of iron to the boiling solution. Black ferrous oxide separates with the vigorous evolution of carbon dioxide. Filter, and evaporate to crystallization.

REACTION: A mixture of ferrous and ferric iodides is formed, which by this decomposition with alkalis gives ferroferric oxide, which is easily filtered off.

TEST: The potassium iodide, in sulphuric acid solution, should not turn starch paper blue, and must show itself free from bromine and chlorine, according to the method of separating the halogens on page 98.

POTASSIUM IODATE, $K_2I_2O_6$.

Literature: Gröger, Zeitschr. angew. Chem. 1894, 13; Erdmann, Lehrbuch der anorganischen Chemie, page 338.

Dissolve 40 grams of pure potassium permanganate in 1 liter of hot water, add 20 grams of potassium iodide dissolved in a small amount of water, heat on a water-bath for 20-30 minutes, and drop in alcohol until the liquid, which is reddened by the excess of the permanganate, is decolorized; then filter. Wash with hot water the precipitate of potassium manganite, which remains on the filter. Then treat the alkaline filtrate with acetic acid, until it reacts distinctly acid, evaporate to about 50 c.c., cool, pour off the mother-liquor from the granular crystals of potassium iodate which separate, wash the crystals repeatedly with strong, pure alcohol, and dry. Yield: 23-25 grams.

REACTION:



Acidifying with acetic acid is necessary; for, otherwise, some alkali persistently adheres to the potassium iodate.

TEST: The potassium iodate must react completely neutral, and an aqueous solution should neither turn yellow, even upon standing for a long time with dilute sulphuric acid, nor react with starch paste. Dissolve in 50 c.c. of water 0.3538 gram of dry pulverized potassium iodate, with the addition of about 2 grams of potassium iodide; add 5 c.c. of dilute hydrochloric acid, and titrate with thiosulphate solution until decolorized. 10.00 c.c. of normal thiosulphate solution are necessary. Pure potassium iodate can also serve as a standard in alkalimetry. For this purpose, dissolve in 50 c.c. of water about 0.4 gram of potassium iodate with 2.5 grams of potassium iodide; add 10 c.c. of nearly normal acid, which is to be standardized; and with the normal thiosulphate, standardized in the manner mentioned above, determine the iodine which has separated. If, for example, in place of 10.00 c.c., only 9.50 c.c. of thiosulphate are used, the factor of the acid solution to be standardized is not 1.00, but 0.950.

IODINE TRICHLORIDE, ICl_3 .

Heat 20 grams of iodine gently in a small retort, the neck of which is connected with a weighed glass

balloon, the balloon being filled with chlorine and closed, but connected with a Kipp apparatus (page 121) for generating chlorine. As soon as the iodine vapors enter the balloon, a rapid absorption of chlorine takes place, and reddish-yellow crystals of the chloride of iodine form and attach themselves firmly to the walls. At the end of the operation, pass dry carbon dioxide through the balloon, in order to drive out the excess of chlorine. Dissolve, in ten times their weight in water, the crystals which cannot be mechanically removed from the balloon, and preserve as a solution of chloride of iodine.

TEST: Iodine trichloride forms an orange-colored crystalline powder of a suffocating odor, and easily dissolves in water to a clear yellow solution. Ammonia should precipitate from a solution the black and very explosive iodide of nitrogen. Chloroform should extract no iodine from an aqueous solution, but should do so after the addition of some stannous chloride. Upon heating, iodine trichloride should be converted into a brown vapor, leaving no residue, the vapors recondensing to an orange-colored sublimate.

IODINE PENTOXIDE, I_2O_5 .

Treat 30 grams of iodine, in a small retort, with 158 grams of water mixed with nitric acid free from the oxides of nitrogen (page 59). The reaction begins

upon shaking, and is hastened by gentle heating. The red vapors that are evolved are driven off by a strong current of air, which is blown in through a glass tube, the tube being made tight in the tubulure by means of asbestos string. In spite of this, a portion of the iodine is always reduced again by the oxides of nitrogen, and, volatilizing, is condensed in a cooled receiver along with any acid that distils. From time to time, stop the heating and, after air has been blown through the distillate, put it back into the retort. Dissolve in a small amount of water the white residue which finally remains, and evaporate to dryness in a porcelain dish on the gas-stove, the anhydride of iodic acid remaining in the form of white crystals. 36-37 grams of anhydride are obtained, the calculated yield being 39.5 grams.

METALLIC MANGANESE, Mn.

Literature: Goldschmidt, *Annalen* **301**, 19; Erdmann, *Lehrbuch der anorganischen Chemie*, page 632.

Gently ignite 900 grams of pure powdered pyrolusite in a fire-clay crucible; and, after cooling, grind up 750 grams of the manganic oxide thus formed, with 250 grams of aluminium powder. Place 2 grams of this mixture at the bottom of the crucible, and cover it with a mixture of $4\frac{1}{2}$ grams of barium peroxide and $\frac{1}{2}$ gram of aluminium powder. Light

the mixture in the crucible by the aid of magnesium ribbon that is wound up and lighted at the end, or better, by a fuse made of a mixture of aluminium and barium peroxide, provided with magnesium ribbon; and, as the reaction progresses, gradually add the prepared mixture to the mass by means of an iron spoon. Too large amounts of the mixture should not be added at a time, so that the reaction does not proceed too vigorously; but small amounts must be added as rapidly as possible, in order to attain the heat necessary to melt the manganese. In order to retain the heat better, the crucible, which soon becomes incandescent on the outside, can be packed in dry sand, or in infusorial earth. Let the covered crucible cool, break it up, and, if the operation has been successful, a dense regulus of metallic manganese is found under a slag consisting essentially of crystallized alumina.

REACTION : $\text{Mn}_2\text{O}_3 + 2\text{Al} = \text{Al}_2\text{O}_3 + 2\text{Mn}$. To start the reaction, a very high temperature is necessary, such as can be produced by burning magnesium or by a mixture of aluminium and barium peroxide. Once started, the process continues spontaneously throughout the whole mass, with the production of much heat. If a crucible were completely filled with the mixture of manganic oxide and aluminium, and heated in a furnace, the reaction would take place with explosive violence. By the gradual addition of

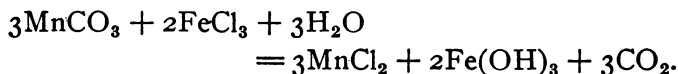
the mixture to the crucible, it is wholly within one's power to regulate the process.

TEST: The great instability that manganese, which contains carbon, shows in the air when in a finely divided or moist condition, should not be exhibited by the metal when prepared by the preceding method. Pure manganese possesses the appearance of bright pig iron, having a red shimmer; it is metallic in lustre and capable of taking a high polish. It is very hard, scratching glass and steel; is brittle, but not magnetic; and, upon heating in the air, it shows a play of color, as in the case of steel. If the heating is continued, it is covered with a brown pulverulent oxide. Manganese is somewhat more difficultly fusible than iron; but it volatilizes at a very high temperature with remarkable ease. Treat a sample with an excess of dilute hydrochloric acid: the metal should dissolve to a clear solution, with the vigorous evolution of hydrogen. A hot solution should give no turbidity, or, at most, a very slight turbidity with an excess of ammonia. By adding ammonium sulphide to the ammoniacal liquid, flesh-colored manganese sulphide precipitates, which, upon heating, gradually turns green.

MANGANOUS CHLORIDE, $\text{MnCl}_2 + 4\text{H}_2\text{O}$.

Evaporate to dryness, in a porcelain dish, the residues from the preparation of chlorine from pyrolusite and hydrochloric acid, and heat the residue, with a small flame, for some time on the gas-stove. Extract with hot water, and precipitate $\frac{1}{10}$ of the filtrate with an excess of sodium carbonate solution. Wash the precipitate a number of times by decantation with water; then add it to the main portion of the liquid, and digest hot, until a filtered test gives, with ammonium sulphide, a pure flesh-colored precipitate which dissolves in acetic acid without leaving a residue. Then filter, and evaporate to crystallization.

REACTION: By heating the dry chlorides, and subsequently heating with water, compounds of the trivalent metals (iron, aluminium) decompose, forming insoluble basic salts. Any remaining portions of such compounds are precipitated by the manganese carbonate which is added:



TEST: The manganous chloride should be tested particularly for the absence of iron, barium, calcium, and magnesium salts.

ANHYDROUS FERROUS CHLORIDE, FeCl_2 .

Erect a retort exactly as in the preparation of ferric chloride (see next preparation), and, after warming it, add as rapidly as possible from a small tube, which has previously been warmed before opening, about 20 grams of anhydrous ferric chloride. Conduct over it a carefully dried current of hydrogen from a Kipp generator. The retort is placed on a gas-stove, but the flame must not be lighted until it is certain that the air is completely driven from the retort. For this purpose, the neck of the retort is stoppered with a cork carrying a tube which is bent upward; and upon the tube a test-tube is inverted, which becomes filled with the gases that are evolved. From time to time, close it with the thumb, and test the explosiveness of the gas by bringing the mouth of the test-tube into contact with a flame. If the gas burns quietly, heat the retort with a moderate-sized flame, whereupon a strong evolution of hydrochloric acid immediately takes place. Absorb the gas in water. The operation is finished when the chloride in the retort has been converted into a white crystalline mass, and the evolution of hydrochloric acid slackens. Cool the contents of the retort in a slow stream of hydrogen, break the retort while it is still warm, and preserve the ferrous chloride which is collected, in the same manner as the anhydrous ferric chloride is preserved.

REACTION: $\text{FeCl}_3 + \text{H} = \text{FeCl}_2 + \text{HCl}$.

TEST: Ferrous chloride forms small white plates, which are tolerably stable in the air, which melt when strongly heated and, at a very high temperature, sublime.

ANHYDROUS FERRIC CHLORIDE, FeCl_3 .

To a tubulated glass retort of about $\frac{3}{4}$ liters capacity, add 50 grams of polished iron wire of about 1 mm. diameter, cut into pieces about 6–8 cm. long. Heat the retort strongly on the gas-stove, and introduce a rapid current of chlorine which has been dried by passing through two wash-bottles containing sulphuric acid. Introduce the chlorine through a rather wide glass tube, which passes through a cork stopper in the tubulure of the retort and ends close to the iron wire. Close the neck of the retort with a perforated stopper; conduct through a rubber tube the chlorine which is evolved, either letting it escape out-of-doors, or absorbing it in an absorption-flask (page 124) by means of caustic soda or alcohol. After the action has continued for 1–2 hours, stop the gas current and the heating, and with dry carbon dioxide drive out the chlorine which fills the retort. Break the retort, while it is still hot, on a large piece of smooth paper, and rapidly collect the ferric chloride. The ferric chloride is easily separated from the fragments of glass, as

well as from any unchanged iron, and is placed in tared test-tubes that have suitable stoppers and that are dry and warm. Immediately seal the test-tubes with a blast-lamp, taking care that the gases from the lamp, which carry aqueous vapor, do not enter the test-tubes.

REACTION: The ferric chloride, which is formed according to the equation $\text{Fe} + 3\text{Cl} = \text{FeCl}_3$, is removed, by its volatility, from the reducing action of the iron, and collects in splendid crystals in the upper portions of the retort.* The process is successful only by the complete exclusion of moisture.

TEST: Ferric chloride forms a dark-colored, compact mass, consisting of small plates that have a green iridescence and an extraordinarily beautiful metallic lustre; it deliquesces rapidly in the air, forming brown drops. It is easily soluble in water, evolving much heat; it dissolves also in alcohol and ether, but with more difficulty in benzene. These solutions are brown in color and react acid.

ANHYDROUS FERROUS BROMIDE, FeBr_2 .

Strongly heat 100 grams of polished iron wire, in a round-bottomed flask on the gas-stove, and slowly dis-

* Ferrous chloride can form only when the current of chlorine is too slow, and it then remains behind with the iron as a fused white mass.

til 100 c.c. of bromine, by means of a water-bath, into the round-bottomed flask. The glass tube conducting the bromine vapors should end close to the bottom of the flask; but, in order that no drop of liquid bromine shall fall upon the hot glass and crack it, it must be protected, either by means of a piece of iron resting on the bottom of the flask, or by means of asbestos. When the reaction has started, the temperature can be moderated somewhat. At the end of the operation, pass dry air or carbon dioxide through the apparatus, and, after breaking the flask, collect and preserve the preparation in the manner described for ferric chloride (page 109).

IRON AMMONIUM ALUM,
 $\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 + 12\text{H}_2\text{O}.$

Dissolve 400 grams of ferrous sulphate in 400 c.c. of water, with the addition of 70 grams of concentrated sulphuric acid. To the boiling-hot solution add concentrated nitric acid (about 120 grams), until a diluted sample gives a pure rust-colored precipitate with ammonia. Evaporate until the solution thickens to a resinous mass, and dilute with water to a specific gravity of 1.317–1.319. Treat 300 grams of this officinal ferric sulphate solution with a solution of 28 grams of ammonium sulphate in 100 grams of water, and let the mixture cool quietly and slowly. Wash

the crystals with cold water, and dry them without heating.

REACTION: By means of the strong concentration, the last traces of nitric acid are removed from the ferric sulphate solution.

TEST: Iron ammonium alum forms amethyst-colored octahedra, the solution of which should show complete freedom from chlorine when treated with silver nitrate.* Determine the amount of iron present, by weighing the residual ferric oxide obtained by ignition in the presence of ammonium nitrate.

PRUSSIAN BLUE.

Wash the residues from the preparation of hydrocyanic acid (page 62), by decantation with water, and treat in a porcelain dish with commercial hydrochloric acid. Prepare a chloride of lime emulsion by suspending chloride of lime in water, and introduce it through a funnel, the stem of which extends to the bottom of the dish, dipping beneath the hydrochloric acid. Stir during the addition, and continue to add the chloride of lime until the mass has become a beautiful blue color and the solution begins to smell of chlorine. Wash the dyestuff by decantation with

* This is essential in the separation of the halogens, when the iron alum is used according to the method described on page 98.

a dilute salt solution, as it does not settle readily out of water.

REACTION: The residue from the hydrocyanic acid consists of the ferrous salt of hydroferrocyanic acid, which is converted into the ferric salt by the oxidizing action of the chloride of lime.

CHLORPLATINIC ACID, H_2PtCl_6
(from residues).

In case platinum residues contain filter-paper and other organic substances, first roast them in a porcelain dish on the gas-stove, then stir them with dilute commercial hydrochloric acid, and completely reduce them by the introduction of zinc bars. Separate mechanically the undissolved zinc, decant, and extract the impure platinum a number of times with hot water and hydrochloric acid. Dissolve the residues in aqua regia, evaporate to a small volume, and precipitate with a concentrated sal ammoniac solution. Filter off the ammonium chlorplatinate and ignite in a porcelain crucible; extract the residual platinum sponge with hydrochloric acid, dissolve it in aqua regia, and evaporate to dryness on a water-bath in a weighed dish, adding a few drops of hydrochloric acid from time to time. Dissolve the residue of pure platinic chloride in 10 parts of water.

TEST: 10 drops of the solution and one drop of

sodium chloride solution, evaporated to a very small volume on a watch-glass, should give a crystalline mass upon cooling quietly. Under the microscope, only well-developed reddish-yellow prisms of sodium chloroplatinate should be seen, and they should not be contaminated by amorphous brown masses (iron, or oxide of nitrogen compounds of platinic chloride).

NATURAL SYSTEM OF THE CHEMICAL ELEMENTS.

H 1.0				He 4.0					
Li 7.0	Be 9.0	B 10.9	C 11.9	N 13.9	O 15.9	F 18.9	Ne 20.3		
Na 22.9	Mg 24.2	Al 26.8	Si 28.2	P 30.8	S 31.8	Cl 35.2	Ar 39.7		
K 38.9	Ca 39.8	Sc 43.7	Ti 47.8	V 51.0	Cr 51.7	Mn 54.6	Fe 55.6	Ni 58.4	Co 59.1
Cu 63.1	Zn 64.9	Ga 69.0	Ge 71.8	As 74.5	Se 78.4	Br 79.3			
Rb 84.8	Sr 87.0	Y 88.3	Zr 90.0	Nb 93.3	Mo 95.3		Ru 100.9	Rh 102.2	Pd 105.6
Ag 107.1	Cd 111.1	In 112.8	Sn 118.0	Sb 119.5	Te 127.0?	I 125.9			
Cs 131.9	Ba 136.4	La 137.6	Ce 139.1	Nd 139.4?	Pr 142.4?	Sa 148.9			
Gd 154.9		Tb 158.8		Er 165.0		Tu 169.4			
Yb 171.7				Ta 181.2	W 182.7		Os 189.6	Ir 191.7	Pt 193.4
Au 195.7	Hg 198.8	Tl 202.6	Pb 205.4	Bi 207.0					
			Th 230.9		U 237.8				

APPENDIX.

SOURCES OF HEAT.

Illuminating-gas is, in general, the best heating material for the laboratory. For evaporating, drying, or distilling at a gentle heat, the gas-stove, shown in the accompanying illustration, is convenient, and is

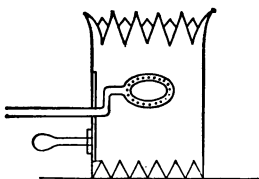


FIGURE 8.—Gas-stove for Gentle Heating.

easily made by any mechanic. It consists of a chimney, 10-15 cm. wide, serrated at the top and bottom, in the middle of which is a gas-pipe, bent in the form of a circle of 4-5 cm. diameter, through which issues a ring of flame through a series of small openings. The temperature is regulated, not only by the supply of gas, but is also capable of such exact regulation by the raising or lowering of the circular flame that it

may, in many cases, advantageously replace a water-bath. Moreover, operations which require a higher but exactly regulated temperature (for example, the preparation of the dry sulphates of the heavy metals, or the separation of silver nitrate from an admixture of cupric nitrate) are extraordinarily facilitated.

For a higher temperature use the so-called Berlin burner, which differs essentially from the one just described, in that the chimney is contracted above in the shape of a cone, the top being closed with wire netting of very fine mesh. The gas and air mixture burns above the wire netting with only a faintly luminous flame. In the simple Berlin burners the gas issues from only one opening in the middle of the burner, 6-7 cm. below the wire netting. Very large heating surfaces may be obtained by passing the gas through a horizontal spiral coil of pipe that is provided with small holes and placed under a wire netting.

An especially suitable form of Bunsen burner is the Dessau burner, which, by placing on a cap containing wire netting, gives a very intimate mixture of the gas with the air, in consequence of which the flame attains a higher temperature and a much greater uniformity. Besides this, the wire netting keeps the flame from striking back, according to the principle of the Davy safety-lamp.

With a relatively small consumption of gas, larger crucibles can be brought to high temperatures by means of the Rössler furnace. Such a furnace, ade-

quate for pyrochemical preparations on a small scale, is shown in the following cross-section.*

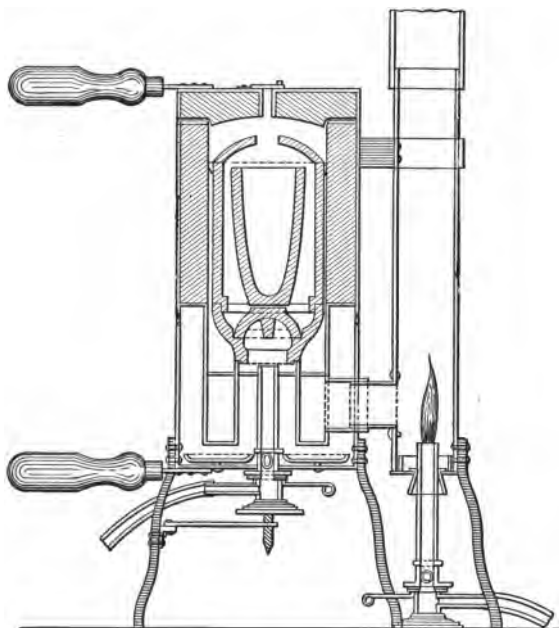


FIGURE 9.—Cross-section of the Rössler Gas-furnace.

The flame completely surrounds the crucible; the gases from the combustion pass out above the crucible through an opening in the highest part of the dome, then move downward, surrounding the whole of that portion of the furnace in which the ignition takes place, which is constructed of fire-clay. The gases

* This furnace will hold a crucible 12 cm. high and 7 cm. upper diameter.

pass into the chimney after they have incidentally warmed the air that is used in the Bunsen burner which does the heating. The chimney may be extended to about 2 meters in length; and in order that the products of combustion may be vigorously drawn up through it, a flame is placed in the chimney. If very high temperatures are necessary, use an acetylene burner in this furnace. This is a Bunsen burner of a somewhat different construction, the mixture of air and acetylene producing substantially higher temperatures than can be obtained with ordinary illuminating gas; but it requires special arrangements, on account of the great rapidity of its explosion-waves, so that the burner will not snap back. Extreme temperatures may also be obtained by means of aluminium powder mixed with metallic oxides, without the use of any furnace (see page 104).

COOLING MIXTURES.

A mixture of three parts of ice and one part of salt serves for cooling between 0° and -20° . Wrap the ice in a cloth and pulverize it to a uniformly coarse powder by means of a hammer. Mix the salt with the powdered ice, and stir thoroughly until the mixture is of a thick consistency, in which an inserted thermometer must show a temperature of at least -20° .

For cooling to a lower temperature, down to -100° , use a mixture of solid carbon dioxide and ether. The solid carbon dioxide is collected by letting the liquefied acid, such as is found in the market, escape into a small bag made of linen or flannel. Place the snow-like mass in a beaker, using a spoon made of horn or wood, and stir ether in with it, until the mixture is of a thick consistency. To test it, place a small test-tube containing chloroform in the mixture: it should rapidly solidify to a crystalline mass (melting-point -83°).

GAS CURRENTS.

The Kipp apparatus serves for generating gases from solid or liquid materials; * if, however, the gas is produced by the interaction of two liquid substances, the Kipp apparatus needs to be supplemented, as shown in Figure 10.

The bulb *B* is filled with an indifferent, porous material, as pumice, upon which one of the reagents drops from *T*, passing through the cock *h*₁ and the mercury trap *E*. The other reagent is in the bulb *A*, and is forced, by the gas which collects, into *C*, when the cock *h*₂ leading to the wash-bottle *W* is closed. *K* is a bulb apparatus filled with a proper liquid for absorption, so that no gas can escape from the upper opening of the Kipp. Figure 11 represents, on a

* For the production of hydrobromic acid, see page 96.

larger scale, the mercury trap *E*, which keeps the gas from passing out through *r* even when, by the sudden

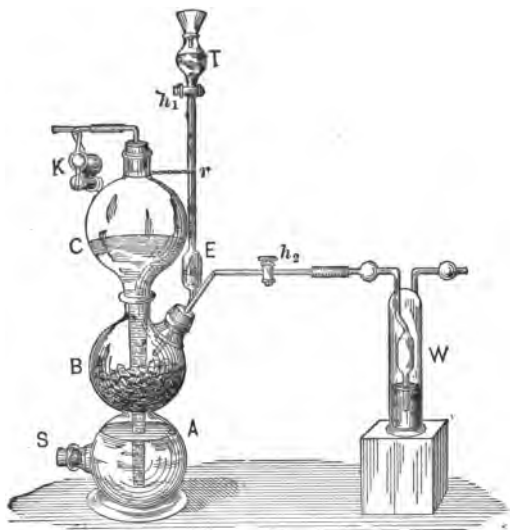


FIGURE 10.—Preparation of Gases by the Interaction of Two Liquid Substances.



FIGURE 11.

closing of h_2 , a considerable pressure arises in the apparatus. In this case, the mercury rises high in the

tube *r* and preserves the equilibrium of the pressure within the apparatus.

In the following table the substances are given from which the various gases are most conveniently prepared.

I. GAS CURRENTS FROM THE KIPP APPARATUS.

Evolution of	Contents of bulb <i>B</i> :	Contents of bulbs <i>A</i> and <i>C</i> :
Chlorine.	Chloride of lime.*	7 liters of commercial hydrochloric acid, 5 liters of water.
Carbon dioxide.	Marble.	1 liter of commercial hydrochloric acid, 1 liter of water.
Methane.	Aluminium carbide.	Lukewarm water
Oxygen.	Chloride of lime.*	1 liter of hydrogen peroxide, 50 c.c. of commercial nitric acid.
Hydrogen sulphide.	Sulphide of iron.	1 liter of commercial hydrochloric acid, 1 liter of water.
Nitric Oxide.	Copper turnings.	Dilute nitric acid.
Hydrogen.	Zinc.	1 liter of sulphuric acid, 4 liters of water.

* Pressed into plates and broken in pieces.

II. GAS CURRENTS FROM THE APPARATUS, FIGURE 10.

Evolution of	Contents of bulb B:	Contents of bulb A:	Reagent flowing in through T:
Acetylene.	Calcium carbide.	Salt solution.	20% sugar solution.
Hydrochloric acid.	Pumice.	Commercial hydrochloric acid.	Concentrated sulphuric acid.
Hydrogen sulphide.	Pumice.	Concentrated sodium sulphide solution.	1 liter of sulphuric acid, 10 liters of water.
Sulphur dioxide.	Pumice.	40% solution of sodium disulphite.	Concentrated sulphuric acid.
Nitric oxide.	Pumice.	Ferrous chloride in hydrochloric acid.	20% solution of sodium nitrite.
Nitrous anhydride.	Pumice.	20% solution of sodium nitrite.	Concentrated sulphuric acid.

If larger amounts of a gas are needed, the frequent emptying of a Kipp apparatus, the contents of which are being very rapidly used up, becomes burdensome; especially in the evolution of chlorine, the pressing and filling in of the material also wastes much time. Consequently, in such cases, it is better to employ the apparatus shown in the accompanying illustration

(Figure 12), which can be filled with solid as well as with liquid substances.

For the evolution of chlorine, place 3.5 kilograms

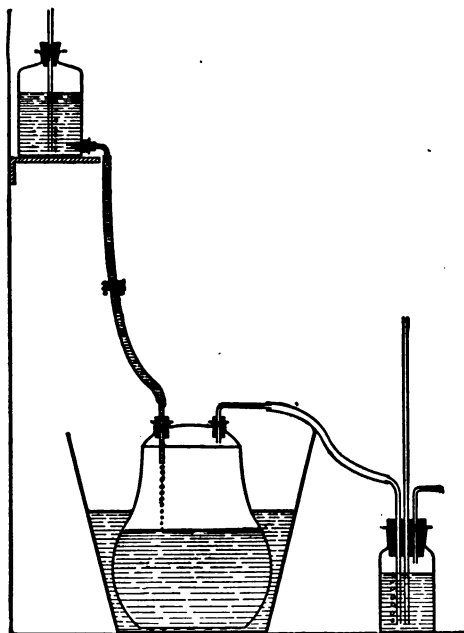


FIGURE 12.—Apparatus for the Evolution of Chlorine.

of chloride of lime, made up to a paste with 2 liters of water, in a porcelain vessel of 15 liters content.*

* These vessels are serviceable for other purposes (for example, they render excellent service in driving over large amounts of substances with steam), and may be obtained in various sizes. The cover is made tight by a moist ring of pasteboard, which is clamped by means of an iron ring provided with screws, which surrounds the whole vessel.

It is necessary to use about 7 liters of commercial hydrochloric acid. Regulate the introduction of the acid by means of a pinch-cock, and control it by the air-bubbles which rise through the hydrochloric acid in the reservoir, the reservoir being fitted with a cork through which a glass tube passes, dipping into the acid.

PURIFICATION OF GAS CURRENTS.—A wash-bottle of the accompanying shape (Figure 13) is suitable

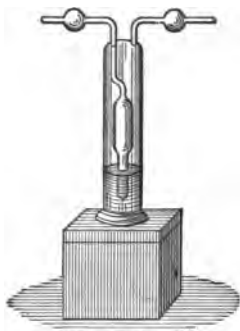


FIGURE 13.—Gas Washing-bottle.

for gas currents, and may contain water, caustic soda, or concentrated sulphuric acid, as may be desired. In this bottle, which is constructed without stoppers, the gas passes through a large number of small openings, and is well broken up in the liquid, the latter, therefore, acting very thoroughly. These glass bottles are provided with a wooden support.

The removal of arsenuretted hydrogen from hydrogen and hydrogen sulphide is best accomplished by

means of iodine.* Pass the gas, which has been washed with water, and consequently contains aqueous vapor, through a tube 30–40 cm. in length, in which 4 grams of iodine are placed in such a manner that thin layers of iodine crystals are separated by glass wool. Wash the gas, as it leaves the tube, with caustic soda and water. Purify chlorine gas from the hydrochloric acid gas which it always contains, by washing it with permanganate solution.†

USE OF LIQUEFIED GASES.—Carbon dioxide, sulphur dioxide, and ammonia, which have recently come into the market in a liquefied form, are specially suitable for use, as they need no further purification. The gas currents obtained from the cylinders containing the liquefied gases, have the great advantage over those obtained from a Kipp apparatus, in that they are almost completely free from air; for example, the carbon dioxide obtained in this way can be used very well for nitrogen determinations by the Dumas method. In order to regulate the gas currents exactly, notwithstanding the very high pressure in the cylinders, a reducing-valve with a pressure-gauge can be used to advantage. It is not advisable to use liquid chlorine and liquid acetylene; for these substances cannot be handled with sufficient safety.

* O. Jakobsen, *Berichte d. d. chem. Ges.* **20**, 1998.

† Hampe, *Chemiker Zeitung* **14**, 1777.

INTRODUCTION OF GASES INTO LIQUIDS.

For the absorption of gases by liquids, flasks or bottles closed air-tight can be conveniently used, the absorption being aided by shaking. This is especially important with poisonous gases and gases having a bad odor, the escape of which into the air would cause considerable annoyance.* If only small amounts of such gases are necessary, they are readily used in aqueous solution. A solution of hydrogen sulphide is troublesome, on account of its instability.



FIGURE 14.—Tube for the Introduction of Gases into Liquids.

Preserve the hydrogen sulphide solution in bottles made of dark glass; by the addition of glycerine, the stability of the solution is increased.† It is of great importance in introducing gases into liquids, as well as in washing them (see page 124), that the gas be

* Gräbe, *Berichte d. d. chem. Ges.* **31**, 2981.

† Shilton, *Chem. News* **62**, 180.

passed into the liquid in the finest possible state of subdivision. For this purpose, the bulb tube, which is perforated with small holes, as represented in Figure 14, is very serviceable.

ABSORPTION OF TROUBLESOME GASES.—For the absorption of troublesome gases, use two of the wash-bottles previously described, or, if larger amounts of gases are to be handled, two Woulff bottles, connected in the manner shown in Figure 15.

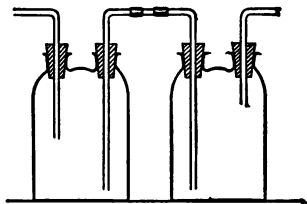


FIGURE 15.—Absorption of Troublesome Gases.

This arrangement offers the advantage that it precludes the sucking back of the absorbing medium by the stoppage of the flow of gas.

ATOMIC WEIGHTS OF THE 74 ELEMENTS.

Under $H = 1$ are found the values calculated with reference to the most recent determinations, and agreeing closely with the tables of F. W. Clarke, which appeared in February, 1899.* Under $O = 16$ are the values adopted by the committee of the German Chemical Society in 1898.† The new elements, such as coronium, polonium, radium, monium, etherion, krypton, neon, xenon, metargon, etc., are not included in this table, as the data concerning them are not sufficiently precise.

		$H = 1.$	$O = 16.$
Aluminium.....	Al	26.8	27.1‡
Antimony.....	Sb	119.5	120
Argon.....	Ar	39.7	40 (?)
Arsenic.....	As	74.5	75
Barium.....	Ba	136.4	137.4
Beryllium.....	Be	9.0	9.1
Bismuth.....	Bi	207.0	208.5 (?)
Boron.....	B	10.9	11
Bromine.....	Br	79.3	79.96
Cadmium.....	Cd	111.5	112
Cæsium.....	Cs	131.9	133
Calcium.....	Ca	39.8	40
Carbon.....	C	11.9	12.00
Cerium.....	Ce	138.3	140
Chlorine.....	Cl	35.2	35.45

* Jour. Am. Chem. Soc. **21**, 213.

† Berichte d. d. chem. Ges. **31**, 2761.

‡ According to the recent researches of Thomson, this number is too high.

		H = 1.	O = 16.
Chromium.....	Cr	51.7	52.1
Cobalt.....	Co	59.1	59
Copper.....	Cu	63.1	63.6
Erbium.....	Er	165.0	166 (?)
Fluorine.....	F	18.9	19
Gadolinium.....	Gd	155.5
Gallium.....	Ga	69.0	70
Germanium.....	Ge	71.8	72
Gold.....	Au	195.7	197.2
Helium.....	He	4.0	4 (?)
Hydrogen.....	H	1.00	1.01 *
Indium.....	In	113.0	114
Iodine.....	I	125.9	126.85
Iridium.....	Ir	191.7	193.0
Iron.....	Fe	55.6	56.0
Lanthanum.....	La	137.6	138
Lead.....	Pb	205.4	206.9
Lithium.....	Li	7.0	7.03
Magnesium.....	Mg	24.1	24.36
Manganese.....	Mn	54.6	55.00
Mercury.....	Hg	198.8	200.3
Molybdenum.....	Mo	95.3	96.0
Neodymium.....	Nd	142.5	144 (?)
Nickel.....	Ni	58.4	58.7 (?)
Niobium.....	Nb	93.0	94
Nitrogen.....	N	13.9	14.04
Osmium.....	Os	189.6	191
Oxygen.....	O	15.9	16.00
Palladium.....	Pd	105.6	106
Phosphorus.....	P	30.8	31.0
Platinum.....	Pt	193.4	194.8
Praseodymium.....	Pr	139.4	140 (?)
Rhodium.....	Rh	102.2	103.0
Rubidium.....	Rb	84.8	85.4
Ruthenium.....	Ru	100.9	101.7
Samarium.....	Sa	149.0	150 (?)
Scandium.....	Sc	43.8	44.1

* More exactly, 1.00762.

		H = 1.	O = 16.
Selenium.....	Se	78.5	79.1
Silicon.....	Si	28.2	28.4
Silver.....	Ag	107.1	107.93
Sodium.....	Na	22.9	23.05
Strontium.....	Sr	87.0	87.6
Sulphur.....	S	31.8	32.06
Tantalum.....	Ta	181.5	183
Tellurium.....	Te	127.0	127
Terbium.....	Tb	158.8
Thallium.....	Tl	202.6	204.4
Thorium.....	Th	230.9	232
Thulium.....	Tu	169.4
Tin.....	Sn	118.0	118.5 (?)
Titanium.....	Ti	47.8	48.1
Tungsten.....	W	183.0	184
Uranium.....	U	237.8	239.5
Vanadium.....	V	51.0	51.2
Ytterbium.....	Yb	171.9	173
Yttrium.....	Y	88.3	89
Zinc.....	Zn	64.9	65.4
Zirconium.....	Zr	90.0	90.6

NOTE.—The question-marks following the atomic weights (O = 16) of the elements A, He, Nd, Pr, and Sa, indicate a doubt as to the elementary nature of these substances. The question-mark is introduced after the atomic weights of Ni, Bi, and Sn, because the Committee of the German Chemical Society questions the accuracy of the decimal figure.

In January, 1900, this same Committee published a revised list of atomic weights (O = 16), the following changes being made: Cd = 112.4; Nd = 143.6; Pr = 140.5; Th = 232.5; Zr = 90.7. The question-marks have also been removed from A, He, Pr, Sa, and Ni.

F. W. Clarke has published his seventh annual report on atomic weights (Journal of the American Chemical Society, February, 1900, page 70). This report discusses the results published on atomic-weight work during 1899. The only notable changes from his previous table (Journal of the American Chemical Society, 1899, page 213) are in the atomic weights of cerium, palladium, and tungsten.



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